9/2495

July 2002

25290-000-0000

Sampling and Analysis Plan
and

# QUALITY ASSURANCE PROJECT PLAN
# J-PIT REDEVELOPMENT SITE
# GARY, INDIANA

*Prepared for*

CITY OF GARY

**Department of Environmental Affairs**

***504 Broadway, Suite 1012***

***Gary, Indiana 46402***

*Prepared by*

**Baker Environmental, Inc.**

***701 East 83rd Avenue, Suite 3***

Merrillville, Indiana 46410

***Prepared by***

Baker

**Baker Environmental, Inc.**

***Merrillville, Indiana***

**Richard Spitaler**
**<RSPITALER@mbakercorp.com>**

To: Jan Pels/R5/USEPA/US@EPA
cc:
Subject: Re: QAPP for Gary Pilot

09/14/02 07:17 PM

Jan

In response to your question, I realized table 3-1 included analysis for polyaromatic hydrocarbons in the footnotes. I believe this is an oversight on our part. Initially there had been discussion to include PAHs as well as other analytes in the J-Pit investigation but it was decided to limit the initial list. It was felt that most potential sources for PAHs would also include SVOC constituents and therefore were not included in initial as part of the initial analytes in order to limit the costs.

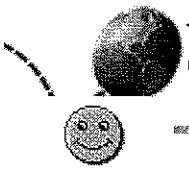
In addition, this agrees with what is stated in the text of the SAP and is also consistent the City of Garys agreement under the voluntary remediation program with the state. (An updated table is attached).

In terms of Baker beginning field work, it is my beleif that we would be able to proceed with the geoprobe portion of the investigation within a couple weeks after recieving approval on the QAPP. I will need to confirm this with the office manager.

Jan if you would like for me to give you a call please let me know what works in you schedule this week.

Thanks
Rick Spitaler

samples 3-1.x



**Jan Pels**
08/23/02 04:39 PM

To: Richard Spitaler <RSPITALER@mbakercorp.com>
cc: mmulligan@ci.gary.in.us
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Rick:

Thanks for the reply!

Regarding the analyses for the metals, I'm still not clear about what list of metals you'll be looking for. The sampling plan (Table 3-2) identifies the 8 RCRA metals (As, Ba, Cd, Cr, Pb, Hg, Se and Ag); however, the reply below includes some additional metals (such as Sb, Ni, V and Zn). The metals list needs to be resolved and corrected such that it's clear what will be analyzed for.

Based on the reply below, we are still missing the 1) lab SOPs for the GFAA sample prep for water water samples, and the 2) lab SOP for GFAA of Cd in waters.

Table 3-1 of the sampling plan needs to reflect that the list of PAHs below will be analyzed for by Method 8270 in soils, and by Method 8310 for waters.

You may want to itemize the list of analytes that are required and attach this list to the sampling plan , similar to what you have below. You may want to do the same for the VOC compounds, so that it's clear whether you're going to look for the entire list of VOCs in Table 1-2 or some sub-set.

Does the lab have a list of soil MDLs for the metals?

I think this is it! Please call or reply if you think we need to discuss this!

Thanks for all of your work on this QAPP.
Jan
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08/22/02 07:19 PM

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Thanks for your assistance. In response to your e-mail, I have attached a copy of the Acronyms list for the QAPP.

In addition, I had previously contacted Jeff Lowe of Sima Labs to answer the questions you had raised in your previous voice mail.

I believe your concerns are addressed in his response presented below ( note these changes are being incorporated in Table 1-2):
Please let me know if these answers are sufficient, they will then be finalized and copies sent to you. Please let me know if you still wish to set up a conference call with Sima to resolve any questions you may still have.

Sincerely, Rick Spitaler

Sima response:

The metals in water will be run as follows:
GFAA- Cd (PQL=0.001 mg/l), As (0.01), Sb (0.02), Se (0.005)
ICP- Pb (0.005), Ag, Ba, Be, Ni, V, Zn

All metals in soil, with the exception of Hg, will be run by ICP with the following PQLs:
Pb (0.25 mg/kg), Cd (0.5), Ag (0.5), Ba (0.5), As, (5), Sb (5), Be (0.5), Ni (1), Se (5), V (1), Zn (1)

All PAHs in water will be run by HPLC (SW8310) with the following PQLs:

| Analyte | PQL, mg/l |
|---|---|
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| Acenaphthylene | 0.0025 |
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| Benzo[b]fluoranthene | 0.0001 |
| Benzo[g,h,i]perylene | 0.0004 |
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| Chrysene | 0.0002 |
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| Fluorene | 0.0005 |
| Indeno[1,2,3cd]pyrene | 0.00025 |
| Naphthalene | 0.0025 |
| Phenanthrene | 0.0002 |
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All PAHs in soil will be run by GC/MS (SW8270C) with the PQLs listed in table 1-2 (or even lower PQLs).

Acronyms.x

**Jan Pels**
09/12/02 03:40 PM

To: Richard Spitaler <RSPITALER@mbakercorp.com>
cc:
Subject: Re: QAPP for Gary Pilot

Rick:

I did find that I do have a copy of the water prep SOP for GFAA metals. It's only the cadmium GFAA SOP that I don't have. Sorry about that!

I have reviewed the tables that you emailed. Table 3-1 is still not clear as to what analytes are being tested for. The table does not include PAHs at all....

Maybe we should discuss these final comments so that we can get the QAPP finalized soon. What does your schedule look like in terms of scheduling field work?

Thanks!
Jan

**Jan Pels**
08/22/02 10:53 AM

To: Richard Spitaler <RSPITALER@mbakercorp.com>
cc: mmulligan@ci.gary.in.us (Mary Mulligan)
Subject: Re: QAPP for Gary Pilot

Rick:

As per our brief discussion yesterday, I took a look at the QAPP, and the following issues still need to be resolved.

- The Acronym List is not included in the copy I have. If you have this page handy, could you FAX it over? Or email it?

- Regarding the list of analytes and PQLs: I am still unclear about what methods are being anticipated to be used for the metals. It appears that some of the metals would be better suited to a GFAA analysis versus the planned ICP method. When I reviewed Sima's method 6010B, which is referenced as the method for the metals (other than mercury), the PQLs that are in the lab's SOP are not those referenced in Table 1-2. For example, lead is listed with a PQL of 0.003 mg/L in Table 1-2, but with an MDL of 0.020 mg/L in the Sima SOP for 6010B- the lab cannot report a PQL lower than their MDL. Since there is no apparent residential standard for lead, the lab could probably do the analysis by ICP, but should use a PQL greater than their MDL.

- Selenium has an MDL of 0.078 mg/L and a PQL of 0.1 mg/L in the Sima SOP,but the GW residential Tier II Screening level is 0.05 mg/L, which is below the lab's MDL. You will probably need to request that the lab do this analysis by GFAA rather than by ICP.

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- Arsenic has an MDL of 0.026 mg/L and a PQL of 0.1 mg/L in the Sima SOP, but the GW residential Tier II Screening level is 0.05 mg/L and a PQL in Table 1-2 of 0.01 mg/L. The lab cannot have a PQL lower than their MDL. You might want to request that arsenic be analyzed by GFAA.

It might be helpful to have a conversation all together with the lab, so that we're all on the same page as far as what analytes are required, what detection limits/methods are appropriate, and to get the issue documented correctly in the QAPP.

Also, we need to have the lab's MDL and PQL for the soil samples for the metals analysis in order to understand whether the methods can meet the State Tier II screening levels for residential soils.

Once we know what methods are appropriate, we can determine whether we have all of the methods in the QAPP.

Please call to discuss this when you have a minute.

Thanks!
Jan
(312) 886-3009

| VOCs by 8260B | |
|---|---|
| Analyte | PQL, |
| 1,1,1,2-Tetrachloroethane | 10 |
| 1,1,1-Trichloroethane | 5 |
| 1,1,2,2-Tetrachloroethane | 5 |
| 1,1,2-Trichloroethane | 5 |
| 1,1-Dichloroethane | 5 |
| 1,1-Dichloroethene | 5 |
| 1,2-Dichloroethane | 5 |
| 1,2-Dichloropropane | 5 |
| 2-Butanone | 10 |
| 2-Hexanone | 10 |
| 4-Methyl-2-Pentanone | 10 |
| Acetone | 50 |
| Acrolein | 100 |
| Acrylonitrile | 100 |
| Benzene | 5 |
| Bromodichloromethane | 5 |
| Bromoform | 5 |
| Bromomethane | 10 |
| Carbon Disulfide | 10 |
| Carbon tetrachloride | 5 |
| Chlorobenzene | 5 |
| Chlorodibromomethane | 5 |
| Chloroethane | 10 |
| Chloroform | 5 |
| Chloromethane | 10 |
| cis-1,2-Dichloroethene | 5 |
| cis-1,3-Dichloropropene | 5 |
| Dibromochloromethane | 5 |
| Dichlorobromomethane | 5 |
| Dichloromethane | 5 |
| Ethylbenzene | 5 |
| m,p-Xylene | 5 |
| Methyl Ethyl Ketone | 10 |
| Methyl Isobutyl Ketone | 5 |
| Methyl-t-Butyl Ether | 10 |
| Methylene chloride | 10 |
| MTBE | 10 |
| o-Xylene | 5 |
| Styrene | 5 |
| tert-Butyl Methyl Ether | 10 |
| Tetrachloroethene | 5 |
| Toluene | 5 |
| trans-1,2-Dichloroethene | 5 |
| trans-1,3-Dichloropropene | 5 |
| Trichloroethene | 5 |
| Trichlorofluoromethane | 5 |
| Vinyl Acetate | 10 |
| Vinyl chloride | 10 |
| 1,1-Dichloropropene | 5 |
| 1,2,3-Trichlorobenzene | 5 |
| 1,2,3-Trichloropropane | 5 |
| 1,2,4-Trichlorobenzene | 5 |
| 1,2,4-Trimethylbenzene | 5 |
| 1,2-Dibromo-3-Chloropropane | 5 |
| 1,2-Dibromoethane | 5 |
| 1,2-Dichlorobenzene | 10 |
| 1,3,5-Trimethylbenzene | 5 |
| 1,3-Dichlorobenzene | 10 |
| 1,3-Dichloropropane | 5 |
| 1,4-Dichlorobenzene | 10 |
| 2,2-Dichloropropane | 5 |
| 2-Chloroethyl vinyl ether | 10 |
| 2-Chlorotoluene | 10 |
| 4-Chlorotoluene | 10 |
| Acetonitrile | 100 |
| Bromobenzene | 5 |
| Cumene | 5 |
| Dibromomethane | 5 |
| Dichlorodifluoromethane | 5 |
| Ethylene Dibromide | 10 |
| Hexachlorobutadiene | 100 |
| Isopropylbenzene | 5 |
| n-Butyl Alcohol | 100 |
| n-Butylbenzene | 10 |
| n-Propylbenzene | 10 |
| Naphthalene | 10 |
| p-Isopropyltoluene | 5 |
| sec-Butylbenzene | 5 |
| tert-Butylbenzene | 5 |
| Total 1,2-Dichloroethene | 5 |
| Total Xylenes | 5 |

| SVOCs by 8270C | |
|---|---|
| Analyte | PQL |
| 1,2,4-Trichlorobenzene | 10 |
| 1,2-Dichlorobenzene | 10 |
| 1,2-Diphenyl-hydrazine | 10 |
| 1,3-Dichlorobenzene | 10 |
| 1,4-Dichlorobenzene | 10 |
| 2,2´-oxybis(1-chloropropane) | 10 |
| 2,4,5-Trichlorophenol | 10 |
| 2,4,6-Trichlorophenol | 10 |
| 2,4-Dichlorophenol | 10 |
| 2,4-Dimethylphenol | 10 |
| 2,4-Dinitrophenol | 50 |
| 2,4-Dinitrotoluene | 10 |
| 2,6-Dichlorophenol | 10 |
| 2,6-Dinitrotoluene | 10 |
| 2-Chloronaphthalene | 10 |
| 2-Chlorophenol | 10 |
| 2-Methyl-4,6-dinitrophenol | 50 |
| 2-Methylnaphthalene | 10 |
| 2-Methylphenol | 10 |
| 2-Nitroaniline | 50 |
| 2-Nitrophenol | 10 |
| 3,3´-Dichlorobenzidine | 50 |
| 3,4-Benzofluoranthene | 10 |
| 3-Methylphenol | 10 |
| 3-Nitroaniline | 50 |
| 3/4-Methylphenol | 10 |
| 4,6-Dinitro-2-methylphenol | 50 |
| 4,6-Dinitro-o-cresol | 50 |
| 4-Bromophenyl phenyl ether | 10 |
| 4-Chloro-3-methylphenol | 20 |
| 4-Chloroaniline | 20 |
| 4-Chlorophenyl phenyl ether | 10 |
| 4-Nitroaniline | 50 |
| 4-Nitrophenol | 50 |
| Acenaphthene | 10 |
| Acenaphthylene | 10 |
| Acetophenone | 10 |
| Aniline | 10 |
| Anthracene | 10 |
| Benzidine | 50 |
| Benzo[a]anthracene | 10 |
| Benzo[a]pyrene | 10 |
| Benzo[b]fluoranthene | 10 |
| Benzo[g,h,i]perylene | 10 |
| Benzo[j]fluoranthene | 10 |
| Benzo[k]fluoranthene | 10 |
| Benzoic acid | 50 |
| Benzyl alcohol | 20 |
| beta-Chloronaphthalene | 10 |
| Bis(2-chloroethoxy)methane | 10 |
| Bis(2-chloroethyl)ether | 10 |
| Bis(2-chloroisopropyl)ether | 10 |
| Bis(2-ethylhexyl)phthalate | 10 |
| Butyl benzyl phthalate | 10 |
| Carbazole | 10 |
| Chrysene | 10 |
| Di(2-ethylhexyl) phthalate | 10 |
| Di-n-butyl phthalate | 10 |
| Di-n-octyl phthalate | 10 |
| Dibenz[a,h]anthracene | 10 |
| Dibenzofuran | 10 |
| Diethyl phthalate | 10 |
| Dimethyl phthalate | 10 |
| Fluoranthene | 10 |
| Fluorene | 10 |
| Hexachlorobenzene | 10 |
| Hexachlorobutadiene | 10 |
| Hexachlorocyclopentadiene | 10 |
| Hexachloroethane | 10 |
| Indeno[1,2,3cd]pyrene | 10 |
| Isophorone | 10 |
| m-Dichlorobenzene | 10 |
| N-Nitrosodi-n-propylamine | 10 |
| N-Nitrosodimethylamine | 10 |
| N-Nitrosodiphenylamine | 10 |
| Naphthalene | 10 |
| Nitrobenzene | 10 |
| o-Chlorophenol | 10 |
| p-Chloro-m-cresol | 20 |
| p-Chloroaniline | 20 |
| p-Cresol | 10 |
| Pentachlorophenol | 50 |
| Phenanthrene | 10 |
| Phenol | 10 |
| Pyrene | 10 |
| Pyridine | 10 |
| Total Cresol | 10 |

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8 RCRA Metals?
OR this list of metals?

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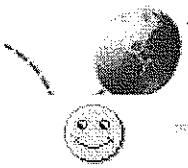
Acronyms.xls



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| Chrysene | 0.0002 |
| Dibenz[a,h]anthracene | 0.0003 |
| Fluoranthene | 0.00025 |
| Fluorene | 0.0005 |
| Indeno[1,2,3cd]pyrene | 0.00025 |
| Naphthalene | 0.0025 |
| Phenanthrene | 0.0002 |
| Pyrene | 0.0005 |

All PAHs in soil will be run by GC/MS (SW8270C) with the PQLs listed in table 1-2 (or even lower PQLs).

Acronyms.xls

**Jan Pels**
09/27/02 04:38 PM

To: Richard Spitaler <RSPITALER@mbakercorp.com>
cc: mmulligan@ci.gary.in.us (Mary Mulligan)
Subject: Re: QAPP for Gary Pilot

Rick:

I got your voicemail. I think the only thing we're really waiting on is the **lab SOP for GFAA analysis of cadmium in water samples.**

I think we can go ahead and get the signature page completed. Can you get the signatures on your end and send me a completed page? If you want an original copy, please complete two copies and send them along (or as many copies as you need). I will sign them and send you back an original or a copy, depending on what you need.

I consider this an approval on our end. The signature page should reflect that the QAPP/SAP is approved before work is started, so please keep this in mind as you get this through the sign-off process.

**It just occurred to me that the cover page and signature page should be modified to reflect that this is a combined "QAPP and Sampling Plan" ! So please title it as such.**

Let's plan to talk next week, if you and Mary are available to briefly discuss the project schedule.

Thanks!
Jan

**Jan Pels**
09/12/02 03:40 PM

To: Richard Spitaler <RSPITALER@mbakercorp.com>
cc:
Subject: Re: QAPP for Gary Pilot

Rick:

I did find that I do have a copy of the water prep SOP for GFAA metals. It's only the cadmium GFAA SOP that I don't have. Sorry about that!

I have reviewed the tables that you emailed. Table 3-1 is still not clear as to what analytes are being tested for. The table does not include PAHs at all....

Maybe we should discuss these final comments so that we can get the QAPP finalized soon. What does your schedule look like in terms of scheduling field work?

Thanks!
Jan

**Richard Spitaler**
**<RSPITALER@mbakercorp.com>**

To: Jan Pels/R5/USEPA/US@EPA
cc:
Subject: Re: QAPP for Gary Pilot

09/14/02 07:17 PM

Jan

In response to your question, I realized table 3-1 included analysis for polyaromatic hydrocarbons in the footnotes. I believe this is an oversight on our part. Initially there had been discussion to include PAHs as well as other analytes in the J-Pit investigation but it was decided to limit the initial list. It was felt that most potential sources for PAHs would also include SVOC constituents and therefore were not included in initial as part of the initial analytes in order to limit the costs.

In addition, this agrees with what is stated in the text of the SAP and is also consistent the City of Garys agreement under the voluntary remediation program with the state. (An updated table is attached).

In terms of Baker beginning field work, it is my beleif that we would be able to proceed with the geoprobe portion of the investigation within a couple weeks after recieving approval on the QAPP. I will need to confirm this with the office manager.

Jan if you would like for me to give you a call please let me know what works in you schedule this week.

Thanks
Rick Spitaler

samples 3-1.x

**Table 3-1**
**Summary Table of Sampling Analysis Program**

| LOCATION | OIL/SEDIMENT | | SURFACE WATER/GROU | |
|---|---|---|---|---|
| | 8 RCRA METALS | VOLATILE ORGANIC COMPOUNDS (VOCs) | SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs) | 8 RCRA METALS |
| *Date Prepared: 4/12/02* | | | | |
| Section 1 | 2 | 7 | 7 | 4 |
| Section 2 | 2 | 8 | 8 | 4 |
| Section 3 | 2 | 8 | 8 | 4 |
| Section 4 | 2 | 7 | 7 | 2 |
| J-Pit Section 5 | 8 | 16 | 16 | 8 |
| Discretionary | 2 | 2 | 2 | 4 |
| QA/QC | 5 | 9 | 9 | 5 |
| Total | 23 | 57 | 57 | 31 |

**NOTE:**

Volatile Organic Compounds (VOCs) analyzed via USEPA SW-846 Method 8260B.

Semi-Volatile Organic Compounds (SVOCs) analyzed via USEPA SW-846 Method 8270A.

Metals (As, Ba, Cd, Cr, Pb, Se, and Ag) are analyzed via USEPA SW-846 Method 6010.

Hg is analyzed via USEPA Method SW-846 method 7471a for solid and 7470a for aqueous.

| DWATER | |
|---|---|
| VOLATILE ORGANIC COMPOUNDS (VOCs) | SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs) |
| | |
| 4 | 4 |
| 4 | 4 |
| 4 | 4 |
| 2 | 2 |
| 8 | 8 |
| 4 | 4 |
| 5 | 5 |
| 31 | 31 |

**Jan Pels**
09/12/02 01:36 PM

To: Richard Spitaler <RSPITALER@mbakercorp.com>
cc:
Subject: Re: QAPP for Gary Pilot

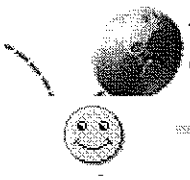
Rick:

The SOPs that are referenced as 'attached here', for metals prep and cadmium analysis are not attached. Could you send these? I'm looking at the tables and will get back with you on those. The discussion about the detection limits for soils is fine.

Thanks!
Jan
Richard Spitaler <RSPITALER@mbakercorp.com>

**Richard Spitaler <RSPITALER@mbakercorp.com>**
09/11/02 08:53 PM

To: Jan Pels/R5/USEPA/US@EPA
cc:
Subject: Re: QAPP for Gary Pilot



**Jan Pels**
08/23/02 04:39 PM

To: Richard Spitaler <RSPITALER@mbakercorp.com>
cc: mmulligan@ci.gary.in.us
Subject: Re: QAPP for Gary Pilot

Rick:

Thanks for the reply!

Regarding the analyses for the metals, I'm still not clear about what list of metals you'll be looking for. The sampling plan (Table 3-2) identifies the 8 RCRA metals (As, Ba, Cd, Cr, Pb, Hg, Se and Ag); however, the reply below includes some additional metals (such as Sb, Ni, V and Zn). The metals list needs to be resolved and corrected such that it's clear what will be analyzed for.

9/12/02
Still missing

Based on the reply below, we are still missing the 1) lab SOPs for the GFAA sample prep for water water samples, and the 2) lab SOP for GFAA of Cd in waters.

Table 3-1 of the sampling plan needs to reflect that the list of PAHs below will be analyzed for by Method 8270 in soils, and by Method 8310 for waters.

You may want to itemize the list of analytes that are required and attach this list to the sampling plan , similar to what you have below. You may want to do the same for the VOC compounds, so that it's clear whether you're going to look for the entire list of VOCs in Table 1-2 or some sub-set.

Does the lab have a list of soil MDLs for the metals? ✓ OK-

I think this is it! Please call or reply if you think we need to discuss this!

Thanks for all of your work on this QAPP.
Jan
Richard Spitaler <RSPITALER@mbakercorp.com>

**Richard Spitaler <RSPITALER@mbakercorp.com>**
08/22/02 07:19 PM

To: Jan Pels/R5/USEPA/US@EPA
cc: mmulligan@ci.gary.in.us
Subject: Re: QAPP for Gary Pilot

Jan:

Thanks for your assistance. In response to your e-mail, I have attached a copy of the Acronyms list for the QAPP.

In addition, I had previously contacted Jeff Lowe of Sima Labs to answer the questions you had raised in your previous voice mail.

I believe your concerns are addressed in his response presented below ( note these changes are being incorporated in Table 1-2):
Please let me know if these answers are sufficient, they will then be finalized and copies sent to you. Please let me know if you still wish to set up a conference call with Sima to resolve any questions you may still have.

Sincerely, Rick Spitaler

Sima response:

The metals in water will be run as follows:
GFAA- Cd (PQL=0.001 mg/l), As (0.01), Sb (0.02), Se (0.005)
ICP- Pb (0.005), Ag, Ba, Be, Ni, V, Zn

All metals in soil, with the exception of Hg, will be run by ICP with the following PQLs:
Pb (0.25 mg/kg), Cd (0.5), Ag (0.5), Ba (0.5), As, (5), Sb (5), Be (0.5), Ni (1), Se (5), V (1), Zn (1)

All PAHs in water will be run by HPLC (SW8310) with the following PQLs:

| Analyte | PQL, mg/l |
|---|---|
| Acenaphthene | 0.005 |
| Acenaphthylene | 0.0025 |
| Anthracene | 0.0001 |
| Benzo[a]anthracene | 0.0001 |
| Benzo[a]pyrene | 0.0002 |
| Benzo[b]fluoranthene | 0.0001 |
| Benzo[g,h,i]perylene | 0.0004 |
| Benzo[k]fluoranthene | 0.0001 |
| Chrysene | 0.0002 |
| Dibenz[a,h]anthracene | 0.0003 |
| Fluoranthene | 0.00025 |
| Fluorene | 0.0005 |
| Indeno[1,2,3cd]pyrene | 0.00025 |
| Naphthalene | 0.0025 |
| Phenanthrene | 0.0002 |
| Pyrene | 0.0005 |

All PAHs in soil will be run by GC/MS (SW8270C) with the PQLs listed in table 1-2 (or even lower PQLs).

Acronyms.xls

**Richard Spitaler <RSPITALER@mbakercorp.com>**

09/11/02 08:53 PM

To: Jan Pels/R5/USEPA/US@EPA
cc:
Subject: Re: QAPP for Gary Pilot

# STATE OF ILLINOIS

## ENVIRONMENTAL PROTECTION AGENCY

# ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

***SIMALABS INTERNATIONAL - MERRILLVILLE***

***250 WEST 84TH DRIVE***

***MERRILLVILLE, IN 46410***

ACCREDITATION NUMBER #100435

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Janet Cruse

Janet Cruse
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 000620
Expiration Date: 01/30/2003
Issued On: 06/28/2002

# *State of Illinois*
# *Environmental Protection Agency*

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The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

***Hazardous and Solid Waste, Inorganic***

*1010*

Ignitability

*1311*

TCLP (Organic and Inorganic)

*1312*

Synthetic Precipitation Leaching Procedure

*6010B*

| | | |
|---|---|---|
| Aluminum | Antimony | Arsenic |
| Barium | Beryllium | Cadmium |
| Calcium | Chromium | Cobalt |
| Copper | Iron | Lead |
| Magnesium | Manganese | Molybdenum |
| Nickel | Potassium | Selenium |
| Silver | Sodium | Strontium |
| Thallium | Vanadium | Zinc |

*7060A*

Arsenic

*7131A*

Cadmium

*7421*

Lead

*7470A*

Mercury

*7471A*

Mercury

*7741A*

Selenium

*7841*

Thallium

*9012A*

Cyanide

*9030B*

Sulfides

*9034*

Sulfides

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***Hazardous and Solid Waste, Inorganic*** *9041A*

Hydrogen Ion (pH)

*9045C*

Hydrogen Ion (pH)

*9066*

Phenolics

***Hazardous and Solid Waste, Organic***

*8081A*

| | | |
|---|---|---|
| 1,2-Dibromo-3-chloropropane (DBCP) | 4,4'-DDD | 4,4'-DDE |
| 4,4'-DDT | alpha-BHC | alpha-Chlordane |
| beta-BHC | Chlordane - not otherwise specified | delta-BHC |
| Dieldrin | Endosulfan I | Endosulfan II |
| Endosulfan sulfate | Endrin | Endrin aldehyde |
| Endrin ketone | gamma-BHC (Lindane) | Heptachlor |
| Heptachlor epoxide | Methoxychlor | Toxaphene |

*8082*

| | | |
|---|---|---|
| PCB-1016 | PCB-1221 | PCB-1232 |
| PCB-1242 | PCB-1248 | PCB-1254 |
| PCB-1260 | | |

*8260B*

| | | |
|---|---|---|
| 1,1,1,2-Tetrachloroethane | 1,1,1-Trichloroethane | 1,1,2,2-Tetrachloroethane |
| 1,1,2-Trichloroethane | 1,1-Dichloroethane | 1,1-Dichloroethene |
| 1,1-Dichloropropene | 1,2,3-Trichlorobenzene | 1,2,4-Trichlorobenzene |
| 1,2,4-Trimethylbenzene | 1,2-Dibromo-3-chloropropane (DBCP) | 1,2-Dibromoethane (EDB) |
| 1,2-Dichlorobenzene | 1,2-Dichloroethane | 1,2-Dichloropropane |
| 1,3,5-Trimethylbenzene | 1,3-Dichlorobenzene | 1,3-Dichloropropane |
| 1,4-Dichlorobenzene | 2-Butanone (Methyl ethyl ketone, MEK) | 2-Chloroethyl vinyl ether |
| 2-Hexanone | 2-Nitropropane | 2-Pentanone |
| 4-Methyl-2-pentanone (Methyl isobutyl ketone, I | Acetone | Acetonitrile |
| Acrolein (Propenal) | Acrylonitrile | Benzene |
| Bromobenzene | Bromodichloromethane | Bromoform |
| Bromomethane | Carbon disulfide | Carbon tetrachloride |
| Chlorobenzene | Chlorodibromomethane (Dibromochloromethan | Chloroethane |
| Chloroform | Chloromethane | cis-1,2-Dichloroethene |
| cis-1,3-Dichloropropene | Dichloromethane (Methylene chloride) | Ethyl acetate |
| Ethylbenzene | Isopropylbenzene | Methyl-t-butyl ether |
| m-Xylene | Naphthalene | n-Butanol |
| n-Butylbenzene | o-Xylene | p-Xylene |
| sec-Butylbenzene | Styrene | tert-Butylbenzene |
| Tetrachloroethene | Toluene | trans-1,2-Dichloroethene |
| trans-1,3-Dichloropropene | Trichloroethene | Trichlorofluoromethane |
| Vinyl acetate | Vinyl chloride | |

*8270C*

| | | |
|---|---|---|
| 1,2,4-Trichlorobenzene | 1,2-Dichlorobenzene | 1,2-Diphenylhydrazine |
| 1,3-Dichlorobenzene | 1,4-Dichlorobenzene | 2,4,5-Trichlorophenol |

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| | | |
|---|---|---|
| ***Hazardous and Solid Waste, Organic*** | *8270C* | 2,4,6-Trichlorophenol |
| 2,4-Dichlorophenol | 2,4-Dimethylphenol | 2,4-Dinitrophenol |
| 2,4-Dinitrotoluene (2,4-DNT) | 2,6-Dichlorophenol | 2,6-Dinitrotoluene (2,6-DNT) |
| 2-Chloronaphthalene | 2-Chlorophenol | 2-Methylnaphthalene |
| 2-Methylphenol | 2-Nitroaniline | 2-Nitrophenol |
| 3,3'-Dichlorobenzidine | 3-Nitroaniline | 4,6-Dinitro-2-methylphenol |
| 4-Bromophenyl phenyl ether | 4-Chloro-3-methylphenol | 4-Chloroaniline |
| 4-Chlorophenyl phenyl ether | 4-Nitroaniline | 4-Nitrophenol |
| Acenaphthene | Acenaphthylene | Acetophenone |
| Aniline | Anthracene | Benzidine |
| Benzo(a)anthracene | Benzo(a)pyrene | Benzo(b)fluoranthene |
| Benzo(g,h,i)perlyene | Benzo(k)fluoranthene | Benzoic acid |
| Benzyl alcohol | Bis(2-chloroethoxy) methane | Bis(2-chloroethyl) ether |
| Bis(2-chloroisopropyl) ether | Bis(2-ethylhexyl) phthalate | Butyl benzyl phthalate |
| Chrysene | Dibenzo(a,h)anthracene | Dibenzofuran |
| Diethyl phthalate | Dimethyl phthalate | Di-n-butyl phthalate |
| Di-n-octyl phthalate | Fluoranthene | Fluorene |
| Hexachlorobenzene | Hexachlorobutadiene | Hexachlorocyclopentadiene |
| Hexachloroethane | Indeno(1,2,3-cd) pyrene | Isophorone |
| m-Cresol (3-Methylphenol) | Naphthalene | Nitrobenzene |
| N-Nitrosodimethylamine | N-Nitrosodi-n-propylamine | N-Nitrosodiphenylamine |
| p-Cresol (4-Methylphenol) | Pentachlorophenol | Phenanthrene |
| Phenol | Pyrene | Pyridine |
| *8310* | | |
| Acenaphthene | Acenaphthylene | Anthracene |
| Benzo(a)anthracene | Benzo(a)pyrene | Benzo(b)fluoranthene |
| Benzo(g,h,i)perylene | Benzo(k)fluoranthene | Chrysene |
| Dibenzo(a,h)anthracene | Fluoranthene | Fluorene |
| Indeno(1,2,3-cd) pyrene | Naphthalene | Phenanthrene |
| Pyrene | | |
| ***Wastewater, Inorganic*** | | |
| *SM2510B,18Ed* | | |
| Specific Conductance | | |
| *SM3500Cr-D,18Ed* | | |
| Chromium VI | | |
| *SM4500CL-B,18Ed* | | |
| Chloride | | |
| *SM4500CN-CE18Ed* | | |
| Cyanide | | |
| *SM4500CN-CG18Ed* | | |
| Cyanide-amenable to chlorination | | |
| *SM4500O-C,18Ed* | | |
| Oxygen - Dissolved | | |
| *SM5210B,18Ed* | | |
| Biochemical Oxygen Demand (BOD) | Carboneous Biochemical Oxygen Demand (CB | |

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---

***Wastewater, Inorganic***

*USEPA110.2*
Color

*USEPA130.2*
Hardness

*USEPA150.1*
Hydrogen Ion (pH)

*USEPA160.1*
Residue (TDS)

*USEPA160.2*
Residue (TSS)

*USEPA160.3*
Residue (Total)

*USEPA160.4*
Residue (Volatile)

*USEPA1664RA*
Oil and Grease

*USEPA170.1*
Temperature

*USEPA200.7*

| | | |
|---|---|---|
| Aluminum | Antimony | Arsenic |
| Barium | Beryllium | Boron |
| Cadmium | Calcium | Chromium |
| Cobalt | Copper | Iron |
| Lead | Magnesium | Manganese |
| Molybdenum | Nickel | Potassium |
| Selenium | Silica | Silver |
| Sodium | Thallium | Tin |
| Vanadium | Zinc | |

*USEPA206.2*
Arsenic

*USEPA213.2*
Cadmium

*USEPA239.2*
Lead

*USEPA245.1*
Mercury

*USEPA270.2*
Selenium

*USEPA279.2*
Thallium

*USEPA310.1*
Alkalinity

*USEPA330.5*

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| | | |
|---|---|---|
| ***Wastewater, Inorganic*** | *USEPA330.5* | Chlorine |
| *USEPA335.2* | | |
| Cyanide | | |
| *USEPA340.2* | | |
| Fluoride | | |
| *USEPA350.1* | | |
| Ammonia | | |
| *USEPA350.2* | | |
| Ammonia | | |
| *USEPA351.3* | | |
| Total Kjeldahl Nitrogen | | |
| *USEPA353.2* | | |
| Nitrate-Nitrite (sum) | | |
| *USEPA354.1* | | |
| Nitrite | | |
| *USEPA365.1* | | |
| Orthophosphate (as P) | | |
| *USEPA365.3* | | |
| Phosphorus | | |
| *USEPA375.4* | | |
| Sulfate | | |
| *USEPA405.1* | | |
| Biochemical Oxygen Demand (BDD) | | |
| *USEPA410.4* | | |
| Chemical Oxygen Demand (COD) | | |
| *USEPA420.2* | | |
| Phenolics | | |
| ***Wastewater, Organic*** | | |
| *USEPA608* | | |
| 4,4'-DDD | 4,4'-DDE | 4,4'-DDT |
| Aldrin | alpha-BHC | beta-BHC |
| Chlordane | delta-BHC | Dieldrin |
| Endosulfan I | Endosulfan II | Endosulfan sulfate |
| Endrin | Endrin aldehyde | gamma-BHC (Lindane) |
| Heptachlor | Heptachlor epoxide | PCB-1016 |
| PCB-1221 | PCB-1232 | PCB-1242 |
| PCB-1248 | PCB-1254 | PCB-1260 |
| Toxaphene | | |
| *USEPA610* | | |
| Acenaphthene | Acenaphthylene | Anthracene |
| Benzo(a)anthracene | Benzo(a)pyrene | Benzo(b)fluoranthene |
| Benzo(g,h,i)perylene | Benzo(k)fluoranthene | Chrysene |
| Dibenzo(a,h)anthracene | Fluoranthene | Fluorene |

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Merrillville, IN 46410

| | | |
|---|---|---|
| ***Wastewater, Organic*** | *USEPA610* | Indeno(1,2,3-cd) pyrene |
| Naphthalene | Phenanthrene | Pyrene |
| *USEPA624* | | |
| 1,1,1-Trichloroethane | 1,1,2,2-Tetrachloroethane | 1,1,2-Trichloroethane |
| 1,1-Dichloroethane | 1,1-Dichloroethene | 1,2-Dichlorobenzene |
| 1,2-Dichloroethane | 1,2-Dichloropropane | 1,3-Dichlorobenzene |
| 1,4-Dichlorobenzene | 2-Chloroethylvinyl ether | Acrylonitrile |
| Benzene | Bromodichloromethane | Bromoform |
| Bromomethane | Carbon tetrachloride | Chlorobenzene |
| Chloroethane | Chloroform | Chloromethane |
| cis-1,3-Dichloropropene | Dibromochloromethane | Dichloromethane (Methylene chloride) |
| Ethylbenzene | Tetrachloroethene | Toluene |
| trans-1,2-Dichloroethene | trans-1,3-Dichloropropene | Trichloroethene |
| Trichlorofluoromethane | Vinyl chloride | |
| *USEPA625* | | |
| 1,2,4-Trichlorobenzene | 1,2-Dichlorobenzene | 1,3-Dichlorobenzene |
| 1,4-Dichlorobenzene | 2,4,6-Trichlorophenol | 2,4-Dichlorophenol |
| 2,4-Dimethylphenol | 2,4-Dinitrotoluene (2,4-DNT) | 2,6-Dinitrotoluene (2,6-DNT) |
| 2-Chloronaphthalene | 2-Chlorophenol | 2-Nitrophenol |
| 3,3'-Dichlorobenzidine | 4-Bromophenyl phenyl ether | 4-Chloro-3-methylphenol |
| 4-Chlorophenyl phenyl ether | 4-Nitrophenol | Acenaphthene |
| Acenaphthylene | Anthracene | Benzidine |
| Benzo(a)anthracene | Benzo(a)pyrene | Benzo(b)fluoranthene |
| Benzo(g,h,i)perylene | Benzo(k)fluoranthene | Benzyl butyl phthalate |
| Bis(2-chloroethoxy) methane | Bis(2-chloroethyl) ether | Bis(2-ethylhexyl) phthalate |
| Chrysene | Dibenzo(a,h)anthracene | Diethyl phthalate |
| Dimethyl phthalate | Di-n-butyl phthalate | Di-n-octyl phthalate |
| Fluoranthene | Fluorene | Hexachlorobenzene |
| Hexachlorobutadiene | Hexachlorocyclopentadiene | Hexachloroethane |
| Indeno(1,2,3-cd) pyrene | Isophorone | Naphthalene |
| Nitrobenzene | N-Nitrosodimethylamine | N-Nitrosodi-n-propylamine |
| N-Nitrosodiphenylamine | Pentachlorophenol | Phenanthrene |
| Phenol | Pyrene | |

SIMALABS International
Data Review Checklist – Metals

Run ID: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| PDS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

- The control is biased high bias yet the analyte is "non-detectable" in the sample.
- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

SIMALABS International
Data Review Checklist – Wet Chemistry

Run ID: _____ Analyte: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD accuracy criteria met? | ☐ Yes ☐ No ☐ NA | |
| MSD / DUP precision criteria met? | ☐ Yes ☐ No ☐ NA | |
| PDS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

- The control is biased high bias yet the analyte is "non-detectable" in the sample.
- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

SIMALABS International
Data Review Checklist – GC

Run ID: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Endrin/DDT breakdown criteria met? (Pesticide analyses only) | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| SURR acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |
| Acceptable confirmation performed on 2nd column? | ☐ Yes ☐ No ☐ NA | |
| Manual integrations appropriately performed and identified? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

- The control is biased high bias yet the analyte is "non-detectable" in the sample.
- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

SIMALABS International
Data Review Checklist – GC/MS

Run ID: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Tune criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ISTD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| SURR acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |
| Manual integrations appropriately performed and identified? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

- The control is biased high bias yet the analyte is "non-detectable" in the sample.
- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

SIMALABS International
Data Review Checklist – HPLC

Run ID: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| SURR acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |
| Manual integrations appropriately performed and identified? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

- The control is biased high bias yet the analyte is "non-detectable" in the sample.
- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

**Table 3-1**
**Summary Table of Sampling Analysis Program**

| LOCATION | SOIL/SEDIMENT | | | SURFACE WATER/GROUNDWATER | | |
|---|---|---|---|---|---|---|
| | METALS | COMPOUNDS (VOCs) | COMPOUNDS (SVOCs) | METALS | COMPOUNDS (VOCs) | COMPOUNDS (SVOCs) |
| *Date Prepared: 4/12/02* | | | | | | |
| Section 1 | 2 | 7 | 7 | 4 | 4 | 4 |
| Section 2 | 2 | 8 | 8 | 4 | 4 | 4 |
| Section 3 | 2 | 8 | 8 | 4 | 4 | 4 |
| Section 4 | 2 | 7 | 7 | 2 | 2 | 2 |
| J-Pit Section 5 | 8 | 16 | 16 | 8 | 8 | 8 |
| Discretionary | 2 | 2 | 2 | 4 | 4 | 4 |
| QA/QC | 5 | 9 | 9 | 5 | 5 | 5 |
| Total | 23 | 57 | 57 | 31 | 31 | 31 |

**NOTE:**

Volatile Organic Compounds (VOCs) analyzed via USEPA SW-846 Method 8260B.
Semi-Volatile Organic Compounds (SVOCs) analyzed via USEPA SW-846 Method 8270A.
Polyaromatic Hydrocarbons (PAHs) analyzed via Method 8270A in soils and by Method 8310 in water.
Metals (As, Ba, Cd, Cr, Pb, Se, and Ag) are analyzed via USEPA SW-846 Method 6010.
Hg is analyzed via USEPA Method SW-846 method 7471a for solid and 7470a for aqueous.

**TABLE 1-2A**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil (1) Nonresidential | | | | | Groundwater(2) Nonresidential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening | PQL(3) | Accuracy (4) | | Precision (4) | Screening | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |
| naphthalene | 10,000.00 | 0.66 | NA | NA | NA | 4.088 | 0.01 | NA | NA | NA |
| acenaphthylene | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| acenaphthene | 10,000.00 | 0.66 | 31 | 78.8 | 24.5 | 6.132 | 0.01 | 28.6 | 88.6 | 41.3 |
| fluorene | 10,000.00 | 0.66 | NA | NA | NA | 4.088 | 0.01 | NA | NA | NA |
| phenanthrene | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| anthracene | 10,000.00 | 0.66 | NA | NA | NA | 30.66 | 0.01 | NA | NA | NA |
| fluoranthene | 10,000.00 | 0.66 | NA | NA | NA | 0.8176 | 0.01 | NA | NA | NA |
| pyrene | 10,000.00 | 0.66 | 26.2 | 87.8 | 21.4 | 3.066 | 0.01 | 25.3 | 103 | 37.7 |
| benzo(a)anthracene* | 103.88 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| chrysene* | 10,000.00 | 0.66 | NA | NA | NA | 0.3918 | 0.01 | NA | NA | NA |
| benzo(b)fluoanthene* | 354.98 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| benzo(k)fluoranthene* | 3,759.12 | 0.66 | NA | NA | NA | 0.0392 | 0.01 | NA | NA | NA |
| benzo(a)pyrene | 69.85 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| indeno(1,2,3-cd)pyrene* | 629.17 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| dibenzo(a,h)anthracene* | 69.86 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| benzo(g,h,i)perylene | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| 3,3'-dichlorobenzidine | 12.86 | 1.3 | NA | NA | NA | 0.02 | 0.02 | NA | NA | NA |
| n-nitroso-di-n-propylamine | 0.66 | 0.66 | 30.7 | 90 | 34 | 0.01 | 0.01 | 15.9 | 119 | 42.7 |
| bis(2-chloroisopropyl)ether | 1.32 | 0.66 | NA | NA | NA | 0.0409 | 0.01 | NA | NA | NA |
| 4-chloroaniline | 1,117.69 | 1.3 | NA | NA | NA | 0.4088 | 0.02 | NA | NA | NA |
| 2-chloronaphthalene | 10,000.00 | 0.66 | NA | NA | NA | 8.176 | 0.01 | NA | NA | NA |
| 2,4-dinitrotoluene | 39.07 | 0.66 | 28.6 | 80 | 12.5 | 0.2044 | 0.01 | 23.5 | 95.4 | 36.5 |
| hexachlorobutadiene | 31.18 | 0.66 | NA | NA | NA | 0.0367 | 0.01 | NA | NA | NA |
| hexachloroethane | 3.31 | 0.66 | NA | NA | NA | 0.0204 | 0.01 | NA | NA | NA |
| isophorone | 256.03 | 0.66 | NA | NA | NA | 3.0105 | 0.01 | NA | NA | NA |
| benzyl alcohol | 4,356.75 | 1.3 | NA | NA | NA | 30.66 | 0.02 | NA | NA | NA |
| bis(2-chloroethyl)ether | 0.66 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| nitrobenzene | 1.73 | 0.66 | NA | NA | NA | 0.0511 | 0.01 | NA | NA | NA |
| 1,2-dichlorobenzene | 10,000.00 | 0.66 | NA | NA | NA | 9.198 | 0.01 | NA | NA | NA |
| 1,3-dichlorobenzene | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| 1,4-dichlorobenzene | 34.67 | 0.66 | 27.8 | 73.3 | 31.1 | 0.1192 | 0.01 | 20.6 | 82 | 39.7 |
| 1,2,4-trichlorobenzene | 1,405.37 | 0.66 | 26.4 | 74.6 | 34.6 | 1.022 | 0.01 | 19.7 | 80.9 | 44.8 |
| hexachlorobenzene | 101.56 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| hexachlorocyclopentadiene | 2.89 | 0.66 | NA | NA | NA | 0.7154 | 0.01 | NA | NA | NA |
| n-nitrosodiphenylamine | 567.8 | 0.66 | NA | NA | NA | 0.5837 | 0.01 | NA | NA | NA |

**TABLE 1-2A**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil (1) Nonresidential | | | | | Groundwater(2) Nonresidential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening | PQL(3) | Accuracy (4) | | Precision (4) | Screening | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |
| benzoic acid | 10,000.00 | 3.3 | NA | NA | NA | 408.8 | 0.05 | NA | NA | NA |
| 2-nitroaniline | 3.3 | 3.3 | NA | NA | NA | 0.05 | 0.05 | NA | NA | NA |
| phenol | 658.78 | 0.66 | 28.1 | 77 | 31.4 | 12.264 | 0.01 | 5 | 46.7 | 70.4 |
| 2-methylphenol | 375.93 | 0.66 | NA | NA | NA | 5.11 | 0.01 | NA | NA | NA |
| 3-methylphenol | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| 4-methylphenol | 427.24 | 0.66 | NA | NA | NA | 5.11 | 0.01 | NA | NA | NA |
| 2-chlorophenol | 11.63 | 0.66 | 29.4 | 75.7 | 32 | 0.511 | 0.01 | 31.3 | 81.3 | 44.3 |
| 2,4-dichlorophenol | 15.12 | 0.66 | NA | NA | NA | 0.3066 | 0.01 | NA | NA | NA |
| 2,4,5-trichlorophenol | 5,507.44 | 0.66 | NA | NA | NA | 10.22 | 0.01 | NA | NA | NA |
| 2,4,6-trichlorophenol | 30.65 | 0.66 | NA | NA | NA | 0.26 | 0.01 | NA | NA | NA |
| pentachlorophenol | 24.95 | 3.3 | 5 | 106 | 12.2 | 0.05 | 0.05 | 5 | 114 | 29.2 |
| 2,4-dinitrophenol | 7.37 | 3.3 | NA | NA | NA | 0.2044 | 0.05 | NA | NA | NA |
| bis(2-ethylhexyl)phthalate | 1,406.25 | 0.66 | NA | NA | NA | 0.2043 | 0.01 | NA | NA | NA |
| butylbenzylphthalate | 10,000.00 | 0.66 | NA | NA | NA | 20.44 | 0.01 | NA | NA | NA |
| di-n-butylphthalate | 6,188.56 | 0.66 | NA | NA | NA | 2.044 | 0.01 | NA | NA | NA |
| diethylphthalate | 10,000.00 | 0.66 | NA | NA | NA | 81.76 | 0.01 | NA | NA | NA |
| de methyl phthalate | 10,000.00 | 0.66 | NA | NA | NA | 1022 | 0.01 | NA | NA | NA |
| di-n-octyl phthalate | 10,000.00 | 0.66 | NA | NA | NA | 2.044 | 0.01 | NA | NA | NA |
| benzene | 4.77 | 0.005 | 85.8 | 120 | 32 | 0.0986 | 0.005 | 74.1 | 119 | 23.7 |
| toluene | 1,000.00 | 0.005 | 80.1 | 126 | 44.7 | 20.44 | 0.005 | 79.6 | 122 | 25.4 |

**TABLE I-2A**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil (1) Nonresidential | | | | | Groundwater(2) Nonresidential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening | PQL(3) | Accuracy (4) | | Precision (4) | Screening | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |
| ethylbenzene | 1,000.00 | 0.005 | 83.8 | 129 | 70.9 | 10.22 | 0.005 | 71.2 | 140 | 25.2 |
| xylenes | 1,000.00 | 0.005 | NA | NA | NA | 204.4 | 0.005 | NA | NA | NA |
| vinyl chloride | 0.13 | 0.01 | 72.2 | 131 | 49.3 | 0.01 | 0.01 | 37.2 | 152 | 31.2 |
| chloroethane | 1,000.00 | 0.01 | 82.6 | 129 | 45 | NA | 0.01 | 5 | 347 | 29.3 |
| 1,1-dichloroethylene | 0.08 | 0.005 | 72.8 | 126 | 36.4 | 0.007 | 0.005 | 56.6 | 124 | 31 |
| 1,1-dichloroethane | 1,000.00 | 0.005 | 86.3 | 110 | 24.1 | 10.22 | 0.005 | 64.3 | 124 | 14.2 |
| 1,2-dichloroethylene (cis) | 102.49 | 0.005 | NA | NA | NA | 1.022 | 0.005 | NA | NA | NA |
| 1,2-dichloroethane | 0.37 | 0.005 | 70.7 | 139 | 41.2 | 0.0314 | 0.005 | 73.3 | 126 | 23.3 |
| trichloroethylene | 25.73 | 0.005 | 82.6 | 116 | 35.3 | 0.26 | 0.005 | 70.7 | 127 | 26.9 |
| 1,1,1-trichloroethane | 1,000.00 | 0.005 | 86.2 | 124 | 24.4 | 9.198 | 0.005 | 72.1 | 129 | 29 |
| 1,1,2-trichloroethane | 1.05 | 0.005 | 72 | 137 | 34.4 | 0.0502 | 0.005 | 75.6 | 123 | 25.9 |
| tetrachloroethylene | 8.01 | 0.005 | 80 | 128 | 29.1 | 0.0561 | 0.005 | 71.7 | 139 | 29.7 |
| 1,1,1,2-tetrachloroethane | 7.24 | 0.005 | NA | NA | NA | 0.11 | 0.005 | NA | NA | NA |
| 1,1,2,2-tetrachloroethane | 0.21 | 0.005 | 77.1 | 145 | 85.2 | 0.0143 | 0.005 | 54.8 | 144 | 27.3 |
| chloroform | 20.33 | 0.005 | 85.3 | 114 | 22.1 | 0.4689 | 0.005 | 75.8 | 116 | 25 |
| acetone | 136.29 | 0.1 | NA | NA | NA | 10.22 | 0.1 | NA | NA | NA |
| 4-methyl-2-pentanone | 407.48 | 0.05 | NA | NA | NA | 5.11 | 0.05 | NA | NA | NA |
| methyl ethyl ketone | 146.24 | 0.1 | NA | NA | NA | 5.11 | 0.1 | NA | NA | NA |
| Aldrin | 0.06 | 0.003 | 50 | 150 | 30 | 0.0002 | 0.00004 | 50 | 150 | 30 |
| gamma-BHC (Lindane) | 0.34 | 0.006 | 50 | 150 | 30 | 0.0022 | 0.00009 | 50 | 150 | 30 |
| chlordane | 4.51 | 0.009 | NA | NA | NA | 0.002 | 0.00014 | NA | NA | NA |
| DDD | 48.34 | 0.007 | 50 | 150 | 30 | 0.0119 | 0.00011 | 50 | 150 | 30 |
| DDE | 80.49 | 0.003 | 50 | 150 | 30 | 0.0084 | 0.00004 | 50 | 150 | 30 |
| DDT | 141.83 | 0.008 | 50 | 150 | 30 | 0.0084 | 0.00012 | 50 | 150 | 30 |
| dieldrin | 0.06 | 0.001 | 50 | 150 | 30 | 0.0002 | 0.00002 | 50 | 150 | 30 |
| endosulfan sulfate | 12 | 0.044 | 50 | 150 | 30 | 0.0051 | 0.00066 | 50 | 150 | 30 |
| endrin | 10.12 | 0.004 | 50 | 150 | 30 | 0.0061 | 0.00006 | 50 | 150 | 30 |
| heptachlor | 0.44 | 0.002 | 50 | 150 | 30 | 0.0006 | 0.00003 | 50 | 150 | 30 |
| heptachlor epoxide | 0.45 | 0.056 | 50 | 150 | 30 | 0.0008 | 0.00083 | 50 | 150 | 30 |
| PCBs (Aroclor 1016) | 4.23 | 0.044 | 51.7 | 133 | 60.3 | 0.0007 | 0.00065 | 30.7 | 153 | 30 |
| lead | NA | 0.5 | NA | NA | NA | NA | 0.003 | 80 | 120 | 20 |
| cadmium | 730 | 0.5 | NA | NA | NA | 0.0511 | 0.005 | 80 | 120 | 20 |
| silver | 7,300.00 | 1 | NA | NA | NA | 0.511 | 0.01 | 85 | 115 | 20 |
| mercury | 87.6 | 0.1 | NA | NA | NA | 0.0061 | 0.0002 | 85 | 115 | 20 |
| chromium vi | 7,300.00 | 1 | 80 | 120 | 20 | 0.511 | 0.01 | 90 | 110 | 20 |

**TABLE 1-2A**

**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil (1) Nonresidential | | | | | Groundwater(2) Nonresidential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening | PQL(3) | Accuracy (4) | | Precision (4) | Screening | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |
| chromium iii | 10,000.00 | 1 | NA | NA | NA | 102.2 | 0.01 | NA | NA | NA |
| barium | 10,000.00 | 20 | NA | NA | NA | 7.154 | 0.2 | 85 | 115 | 20 |
| arsenic | 438 | 1 | NA | NA | NA | 0.05 | 0.01 | 80 | 120 | 20 |
| antimony | 584 | 6 | NA | NA | NA | 0.06 | 0.06 | 80 | 120 | 20 |
| beryllium | 118.6 | 0.5 | NA | NA | NA | 0.005 | 0.005 | 88 | 115 | 20 |
| cyanide | 10,000.00 | 0.125 | NA | NA | NA | 2.044 | 0.01 | 90 | 110 | 20 |
| nickel | 10,000.00 | 4 | NA | NA | NA | 2.044 | 0.04 | 85 | 115 | 20 |
| selenium | 7,300.00 | 0.5 | NA | NA | NA | 0.511 | 0.005 | 80 | 120 | 20 |
| vanadium | 10,000.00 | 5 | NA | NA | NA | 0.7154 | 0.05 | 85 | 115 | 20 |
| zinc | 10,000.00 | 2 | NA | NA | NA | 30.66 | 0.02 | 85 | 115 | 20 |

Footnotes:

1. Data Quality Objectives for subsurface soil are taken from Indian Department of Environmetal Management, Summary of Health-Based Criteria for Subsurface Soils (Nonresidential Land-Use Scenario), in the Resource Guide for the I Voluntary Remediation Program, Appendix F, Table 10 (July 1996).

2. Data Quality Objectives for subsurface soil are taken from Indian Department of Environmetal Management, Summary of Health-Based Criteria for Groundwater (Nonresidential Land-Use Scenario), in the Resource Guide for the Indi Voluntary Remediation Program, Appendix F, Table 8 (July 1996).

3. PQL - Practical Quantitation Limit, based on EPA SW-846, 1986 for GC/MS.

4. Subject to change based on current laboratory in-house acceptance limits.

5. Subject to change based on vendor supplied acceptance limits for a solid matrix standard.

NA- Not Available since organic constituents are not spiked and controlled. List of spiked constituents are defined in method, however, the spike list is subject to change. Specific accuracy and precision criteria are not available for meta

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil (1) Residential | | | | | Groundwater(2) Residential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level | PQL(3) | Accuracy (4) | | Precision (4) | Screening Level | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |
| **Semivolatile Organic Compounds** | | | | | | | | | | |
| naphthalene | 1,761.785 | 0.660 | NA | NA | NA | 1.21600 | 0.01000 | NA | NA | NA |
| acenaphthylene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA | NA |
| acenaphthene | 10,000.000 | 0.660 | 17.1 | 89.1 | 24.5 | 1.82400 | 0.01000 | 14.5 | 96.6 | 41.3 |
| fluorene | 8,838.641 | 0.660 | 29 | 156 | 46 | 1.21600 | 0.01000 | NA | NA | NA |
| phenanthrene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA | NA |
| anthracene | 10,000.000 | 0.660 | NA | NA | NA | 9.12000 | 0.01000 | NA | NA | NA |
| fluoranthene | 2,305.040 | 0.660 | NA | NA | NA | 0.24320 | 0.01000 | NA | NA | NA |
| pyrene | 10,000.000 | 0.660 | 5 | 116 | 21.4 | 0.91200 | 0.01000 | 14.3 | 107 | 37.7 |
| benzo(a)anthracene | 103.881 | 0.660 | NA | NA | NA | 0.00010 | 0.01000 | NA | NA | NA |
| chrysene | 379.273 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA | NA |
| benzo(b)fluoranthene | 354.977 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA | NA |
| benzo(k)fluoranthene | 501.638 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA | NA |
| benzo(a)pyrene | 69.849 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA | NA |
| indeno(1,2,3-cd)pyrene | 629.166 | 0.660 | NA | NA | NA | 0.00040 | 0.01000 | NA | NA | NA |
| dibenzo(a,h)anthracene | 69.863 | 0.660 | NA | NA | NA | 0.00030 | 0.01000 | NA | NA | NA |
| benzo(g,h,i)perylene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA | NA |
| 3,3'-dichlorobenzidine | 12.865 | 1.300 | NA | NA | NA | 0.02000 | 0.02000 | NA | NA | NA |
| n-nitroso-di-n-propylamine | 0.660 | 0.660 | 9.25 | 109 | 34 | 0.01000 | 0.01000 | 6.65 | 116 | 42.7 |
| bis(2-chloroisopropyl)ether | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| 4-chloroaniline | 186.921 | 1.300 | NA | NA | NA | 0.12160 | 0.02000 | NA | NA | NA |
| 2-chloronaphthalene | 10,000.000 | 0.660 | NA | NA | NA | 2.43200 | 0.01000 | NA | NA | NA |
| 2,4-dinitrotoluene4 | 6.535 | 0.660 | 12.5 | 83.4 | 29.7 | 0.06080 | 0.01000 | 18.7 | 96.2 | 36.5 |
| hexachlorobutadiene | 6.777 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| hexachloroethane | 1.153 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| isophorone | 1.433 | 0.660 | NA | NA | NA | 0.08947 | 0.01000 | NA | NA | NA |
| benzyl alcohol | 728.618 | 1.300 | NA | NA | NA | 9.12000 | 0.02000 | NA | NA | NA |
| bis(2-chloroethyl)ether | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| nitrobenzene | 0.660 | 0.660 | NA | NA | NA | 0.01520 | 0.01000 | NA | NA | NA |
| 1,2-dichlorobenzene | 2,524.230 | 0.660 | NA | NA | NA | 0.60000 | 0.01000 | NA | NA | NA |
| 1,3-dichlorobenzene | NA | 0.660 | NA | NA | NA | 0.60000 | 0.01000 | NA | NA | NA |
| 1,4-dichlorobenzene | 0.897 | 0.660 | 5 | 93.1 | 31.1 | 0.07500 | 0.01000 | 12.1 | 93.1 | 39.7 |
| 1,2,4-trichlorobenzene | 235.033 | 0.660 | 5 | 86.2 | 34.6 | 0.07000 | 0.01000 | 6.56 | 93.1 | 44.8 |
| hexachlorobenzene4 | 101.564 | 0.660 | NA | NA | NA | 0.00100 | 0.01000 | NA | NA | NA |
| hexachlorocyclopentadiene | 2.891 | 0.660 | NA | NA | NA | 0.05000 | 0.01000 | NA | NA | NA |

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil (1) Residential | | | | | Groundwater(2) Residential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level | PQL(3) | Accuracy (4) | | Precision (4) | Screening Level | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |
| n-nitrosodiphenylamine | 3.177 | 0.660 | NA | NA | NA | 0.01735 | 0.01000 | NA | NA | NA |
| benzoic acid | 10,000.000 | 3.300 | NA | NA | NA | 121.60000 | 0.05000 | NA | NA | NA |
| 2-nitroaniline | 3.300 | 3.300 | NA | NA | NA | 0.05000 | 0.05000 | NA | NA | NA |

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil (1) Residential | | | | | Groundwater(2) Residential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level | PQL(3) | Accuracy (4) | | Precision (4) | Screening Level | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |
| phenol | 110.173 | 0.660 | 10.8 | 87.8 | 31.4 | 3.64800 | 0.01000 | 5 | 70.4 | 55 |
| 2-methylphenol | 62.871 | 0.660 | NA | NA | NA | 1.52000 | 0.01000 | NA | NA | NA |
| 3-methylphenol | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA | NA |
| 4-methylphenol | 71.452 | 0.660 | NA | NA | NA | 1.52000 | 0.01000 | NA | NA | NA |
| 2-chlorophenol | 1.945 | 0.660 | 19.6 | 76.5 | 32 | 0.15200 | 0.01000 | 5 | 106 | 44.3 |
| 2,4-dichlorophenol4 | 2.528 | 0.660 | NA | NA | NA | 0.09120 | 0.01000 | NA | NA | NA |
| 2,4,5-trichlorophenol | 921.059 | 0.660 | NA | NA | NA | 3.04000 | 0.01000 | NA | NA | NA |
| 2,4,6-trichlorophenol4 | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| pentachlorophenol | 24.947 | 3.300 | 5 | 91.3 | 12.2 | 0.00100 | 0.05000 | 5 | 135 | 29.2 |
| 2,4-dinitrophenol | 3.300 | 3.300 | NA | NA | NA | 0.06080 | 0.05000 | NA | NA | NA |
| bis(2-ethylhexyl)phthalate4 | 16.427 | 0.660 | NA | NA | NA | 0.00600 | 0.01000 | NA | NA | NA |
| butylbenzylphthalate | 10,000.000 | 0.660 | NA | NA | NA | 0.10000 | 0.01000 | NA | NA | NA |
| di-n-butylphthalate4 | 1,034.967 | 0.660 | NA | NA | NA | 0.60800 | 0.01000 | NA | NA | NA |
| diethylphthalate | 10,000.000 | 0.660 | NA | NA | NA | 24.32000 | 0.01000 | NA | NA | NA |
| de methyl phthalate | 10,000.000 | 0.660 | NA | NA | NA | 304.00000 | 0.01000 | NA | NA | NA |
| di-n-octyl phthalate | 2,318.850 | 0.660 | NA | NA | NA | 0.60800 | 0.01000 | NA | NA | NA |
| **Volatile Organic Compounds** | | | | | | | | | | |
| benzene | 0.059 | 0.005 | 54.3 | 134 | 32 | 0.00500 | 0.00500 | 60.6 | 130 | 23.7 |
| toluene | 278.926 | 0.005 | 45.3 | 147 | 44.7 | 1.00000 | 0.00500 | 69.7 | 130 | 25.4 |
| ethylbenzene | 834.372 | 0.005 | 33 | 161 | 70.9 | 0.70000 | 0.00500 | 60.6 | 144 | 25.2 |
| xylenes | 1,000.000 | 0.005 | NA | NA | NA | 10.00000 | 0.00500 | NA | NA | NA |
| vinyl chloride | 0.129 | 0.010 | 21.5 | 174 | 49.3 | 0.00200 | 0.01000 | 33 | 151 | 31.2 |
| chloroethane | 1,000.000 | 0.010 | 0 | 439 | 45 | 23.16075 | 0.01000 | 0 | 357 | 29.3 |
| 1,1-dichloroethylene | 0.084 | 0.005 | 40.5 | 140 | 36.4 | 0.00700 | 0.00500 | 50.1 | 125 | 31 |
| 1,1-dichloroethane | 40.074 | 0.005 | 60.5 | 134 | 24.1 | 0.64000 | 0.00500 | 68 | 119 | 14.2 |
| 1,2-dichloroethylene (cis) | 17.140 | 0.005 | NA | NA | NA | 0.07000 | 0.00500 | NA | NA | NA |
| 1,2-dichloroethane | 0.025 | 0.005 | 55.9 | 149 | 41.2 | 0.00500 | 0.00500 | 67.7 | 133 | 23.3 |
| trichloroethylene | 0.076 | 0.005 | 41.8 | 145 | 35.3 | 0.00500 | 0.00500 | 48.6 | 139 | 26.9 |
| 1,1,1-trichloroethane | 229.642 | 0.005 | 51.5 | 149 | 24.4 | 0.20000 | 0.00500 | 63 | 136 | 29 |
| 1,1,2-trichloroethane | 0.035 | 0.005 | 49.2 | 147 | 34.4 | 0.00500 | 0.00500 | 40.6 | 165 | 25.9 |
| tetrachloroethylene | 0.227 | 0.005 | 51.8 | 139 | 29.1 | 0.00500 | 0.00500 | 60.1 | 144 | 29.7 |
| 1,1,1,2-tetrachloroethane | 0.076 | 0.005 | NA | NA | NA | 0.00500 | 0.00500 | NA | NA | NA |
| 1,1,2,2-tetrachloroethane | 0.044 | 0.005 | 24.9 | 201 | 85.2 | 0.00500 | 0.00500 | 46.8 | 168 | 22.3 |
| chloroform | 2.082 | 0.005 | 59.8 | 126 | 22.1 | 0.10000 | 0.00500 | 71.2 | 120 | 25 |
| acetone | 22.793 | 0.100 | NA | NA | NA | 3.04000 | 0.10000 | NA | NA | NA |

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Snil (1) Residential | | | | | Groundwater(2) Residential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level | PQL(3) | Accuracy (4) | | Precision (4) | Screening Level | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |
| 4-methyl-2-pentanone | 68.147 | 0.050 | NA | NA | NA | 1.52000 | 0.05000 | NA | NA | NA |
| methyl ethyl ketone | 11.620 | 0.100 | NA | NA | NA | 0.91772 | 0.10000 | NA | NA | NA |
| **Pesticides** | | | | | | | | | | |
| Aldrin | 0.007 | 0.003 | 34 | 132 | 43 | 0.00004 | 0.00004 | 40 | 120 | 22 |
| gamma-BHC (Lindane) | 0.010 | 0.006 | 46 | 127 | 50 | 0.00020 | 0.00009 | 56 | 123 | 15 |
| chlordane | 4.512 | 0.009 | NA | NA | NA | 0.00200 | 0.00014 | NA | NA | NA |
| DDD | 0.270 | 0.007 | NA | NA | NA | 0.00035 | 0.00011 | NA | NA | NA |
| DDE | 0.450 | 0.003 | NA | NA | NA | 0.00025 | 0.00004 | NA | NA | NA |
| DDT | 0.794 | 0.008 | NA | NA | NA | 0.00025 | 0.00012 | NA | NA | NA |
| dieldrin | 0.003 | 0.001 | 31 | 134 | 38 | 0.00002 | 0.00002 | 52 | 126 | 18 |
| endosulfan sulfate | 2.007 | 0.044 | NA | NA | NA | 0.00152 | 0.00066 | NA | NA | NA |
| endrin | 1.939 | 0.004 | 42 | 139 | 45 | 0.00200 | 0.00006 | 56 | 121 | 21 |
| heptachlor | 0.221 | 0.002 | 35 | 130 | 31 | 0.00040 | 0.00003 | 40 | 131 | 20 |
| heptachlor epoxide | 0.450 | 0.056 | NA | NA | NA | 0.00020 | 0.00083 | NA | NA | NA |
| **PCBs** | | | | | | | | | | |
| PCBs (Aroclor 1016) | 4.226 | 0.044 | 70 | 130 | NA | 0.00050 | 0.00065 | 70 | 130 | NA |
| **Metals** | | | | | | | | | | |
| lead | NA | 0.500 | 70 | 130 | 20 | NA | 0.00300 | 75 | 125 | 20 |
| cadmium | 730.000 | 0.500 | 70 | 130 | 20 | 0.00500 | 0.00500 | 75 | 125 | 20 |
| silver | 7,300.000 | 1.000 | 70 | 130 | 20 | 0.15200 | 0.01000 | 75 | 125 | 20 |
| mercury | 87.600 | 0.100 | 70 | 130 | 20 | 0.00200 | 0.00020 | 75 | 125 | 20 |
| chromium vi | 7,300.000 | 1.000 | 70 | 130 | 20 | 0.10000 | 0.01000 | 75 | 125 | 20 |
| chromium iii | 10,000.000 | 1.000 | 70 | 130 | 20 | 0.10000 | 0.01000 | 75 | 125 | 20 |
| barium | 10,000.000 | 20.000 | 70 | 130 | 20 | 2.00000 | 0.20000 | 75 | 125 | 20 |
| arsenic | 438.000 | 1.000 | 70 | 130 | 20 | 0.05000 | 0.01000 | 75 | 125 | 20 |
| antimony | 584.000 | 6.000 | 70 | 130 | 20 | 0.00600 | 0.06000 | 75 | 125 | 20 |
| beryllium | 118.605 | 0.500 | 70 | 130 | 20 | 0.00400 | 0.00500 | 75 | 125 | 20 |
| cyanide | 10,000.000 | 0.125 | 70 | 130 | 20 | 0.20000 | 0.01000 | 75 | 125 | 20 |
| nickel | 10,000.000 | 4.000 | 70 | 130 | 20 | 0.10000 | 0.04000 | 75 | 125 | 20 |
| selenium | 7,300.000 | 0.500 | 70 | 130 | 20 | 0.05000 | 0.00500 | 75 | 125 | 20 |
| vanadium | 10,000.000 | 5.000 | 70 | 130 | 20 | 0.21280 | 0.05000 | 75 | 125 | 20 |
| zinc | 10,000.000 | 2.000 | 70 | 130 | 20 | 9.12000 | 0.02000 | 75 | 125 | 20 |

Footnotes:

1- Tier II screening levels for subsurface soil are taken from Indiana Department of Environmental Management, Summary of Health-Based Criteria for Subsurface Soils (Nonresidential Land-Use Scenario), in the Resource Guide for the Indiana Voluntary Remed

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil (1) Residential | | | | | Groundwater(2) Residential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level | PQL(3) | Accuracy (4) | | Precision (4) | Screening Level | PQL(3) | Accuracy (5) | | Precision (5) |
| | | | LCL | UCL | RPD | | | LCL | UCL | RPD |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % | % |

2- Tier II screening levels for groundwater are taken from Indiana Department of Environmental Management, Summary of Health-Based Criteria for groundwater (Non-residential Land-use Scenario), in the Resource Guide for the Indiana Voluntary Remediation

3- PQL - Practical Quantitation Limit, based on EPA SW-846, 1986 for GC/MS.

4- Compounds are not spiked in Method 8270.
NA-Not Applicable

| ELEMENT | AQUEOUS | SOLID | |
|---|---|---|---|
| | | MDL, mg/l | MDL, mg/kg |
| As (ICP) | 0.035 | | 3.5 |
| As (GFAA) | 0.005 | | 0.5 |
| Ba (ICP) | 0.001 | | 0.1 |
| Cd (ICP) | 0.002 | | 0.2 |
| Cd (GFAA) | 0.00003 | 0.003 | |
| Cr (ICP) | 0.003 | | 0.3 |
| Pb (ICP) | 0.0025 | 0.2 | |
| Pb (GFAA) | 0.002 | | 0.2 |
| Se (ICP) | 0.036 | | 3.6 |
| Se (GFAA) | 0.002 | | 0.2 |
| Ag (ICP) | 0.003 | | 0.3 |
| Hg (CVAA) | 0.00003 | 0.003 | |

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil [1] Residential | | | | | Groundwater [2] Residential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level (mg/kg) | PQL [3] (mg/kg) | Accuracy [4] LCL % | Accuracy [4] UCL % | Precision [4] RPD % | Screening Level (mg/l) | PQL [3] (mg/l) | Accuracy [5] LCL % | Accuracy [5] UCL % | Precision [5] RPD % |
| **Pesticides** | | | | | | | | | | |
| Aldrin | 0.007 | 0.003 | 34 | 132 | 43 | 0.00004 | 0.00004 | 40 | 120 | 22 |
| gamma-BHC (Lindane) | 0.010 | 0.006 | 46 | 127 | 50 | 0.00020 | 0.00009 | 56 | 123 | 15 |
| chlordane | 4.512 | 0.009 | NA | NA | NA | 0.00200 | 0.00014 | NA | NA | NA |
| DDD | 0.270 | 0.007 | NA | NA | NA | 0.00035 | 0.00011 | NA | NA | NA |
| DDE | 0.450 | 0.003 | NA | NA | NA | 0.00025 | 0.00004 | NA | NA | NA |
| DDT | 0.794 | 0.008 | NA | NA | NA | 0.00025 | 0.00012 | NA | NA | NA |
| dieldrin | 0.003 | 0.001 | 31 | 134 | 38 | 0.00002 | 0.00002 | 52 | 126 | 18 |
| endosulfan sulfate | 2.007 | 0.044 | NA | NA | NA | 0.00152 | 0.00066 | NA | NA | NA |
| endrin | 1.939 | 0.004 | 42 | 139 | 45 | 0.00200 | 0.00006 | 56 | 121 | 21 |
| heptachlor | 0.221 | 0.002 | 35 | 130 | 31 | 0.00040 | 0.00003 | 40 | 131 | 20 |
| heptachlor epoxide | 0.450 | 0.056 | NA | NA | NA | 0.00020 | 0.00083 | NA | NA | NA |
| **PCBs** | | | | | | | | | | |
| PCBs (Aroclor 1016) | 4.226 | 0.044 | 70 | 130 | NA | 0.00050 | 0.00065 | 70 | 130 | NA |
| **Metals** | | | | | | | | | | |
| lead | NA | 0.500 | 70 | 130 | 20 | NA | 0.00300 | 75 | 125 | 20 |
| cadmium | 730.000 | 0.500 | 70 | 130 | 20 | 0.00500 | 0.00500 | 75 | 125 | 20 |
| silver | 7,300.000 | 1.000 | 70 | 130 | 20 | 0.15200 | 0.01000 | 75 | 125 | 20 |
| mercury | 87.600 | 0.100 | 70 | 130 | 20 | 0.00200 | 0.00020 | 75 | 125 | 20 |
| chromium vi | 7,300.000 | 1.000 | 70 | 130 | 20 | 0.10000 | 0.01000 | 75 | 125 | 20 |
| chromium iii | 10,000.000 | 1.000 | 70 | 130 | 20 | 0.10000 | 0.01000 | 75 | 125 | 20 |
| barium | 10,000.000 | 20.000 | 70 | 130 | 20 | 2.00000 | 0.20000 | 75 | 125 | 20 |
| arsenic | 438.000 | 1.000 | 70 | 130 | 20 | 0.05000 | 0.01000 | 75 | 125 | 20 |
| antimony | 584.000 | 6.000 | 70 | 130 | 20 | 0.00600 | 0.06000 | 75 | 125 | 20 |
| beryllium | 118.605 | 0.500 | 70 | 130 | 20 | 0.00400 | 0.00500 | 75 | 125 | 20 |
| cyanide | 10,000.000 | 0.125 | 70 | 130 | 20 | 0.20000 | 0.01000 | 75 | 125 | 20 |
| nickel | 10,000.000 | 4.000 | 70 | 130 | 20 | 0.10000 | 0.04000 | 75 | 125 | 20 |
| selenium | 7,300.000 | 0.500 | 70 | 130 | 20 | 0.05000 | 0.00500 | 75 | 125 | 20 |
| vanadium | 10,000.000 | 5.000 | 70 | 130 | 20 | 0.21280 | 0.05000 | 75 | 125 | 20 |
| zinc | 10,000.000 | 2.000 | 70 | 130 | 20 | 9.12000 | 0.02000 | 75 | 125 | 20 |

Footnotes:

1- Tier II screening levels for subsurface soil are taken from Indiana Department of Environmental Management, Summary of Health-Based Criteria for Subsurface Soils (Nonresidential Land-Use Scenario)n the Resource Guide for the Indiana Voluntary Remediation Program, Appendix F, Table 10 (July 1996).

2- Tier II screening levels for groundwater are taken from Indiana Department of Environmental Management, Summary of Health-Based Criteria for groundwater (Non-residential Land-use Scenario)n the Resource Guide for the Indiana Voluntary Remediation Program Appendix F, Table 8 (July 1996).

3- PQL - Practical Quantitation Limit, based on EPA SW-846, 1986 for GC/MS.

4- Compounds are not spiked in Method 8270.

NA-Not Applicable

| Constituent | Subsurface | | | | | roundwater(2 |
|---|---|---|---|---|---|---|
| | Screening | PQL(3) | Accuracy (4) | | Precision (4) | Screening |
| | | | LCL | UCL | RPD | |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) |
| naphthalene | 10,000.00 | 0.66 | NA | NA | NA | 4.088 |
| acenaphthylene | NA | 0.66 | NA | NA | NA | NA |
| acenaphthene | 10,000.00 | 0.66 | 31 | 78.8 | 24.5 | 6.132 |
| fluorene | 10,000.00 | 0.66 | NA | NA | NA | 4.088 |
| phenanthrene | NA | 0.66 | NA | NA | NA | NA |
| anthracene | 10,000.00 | 0.66 | NA | NA | NA | 30.66 |
| fluoranthene | 10,000.00 | 0.66 | NA | NA | NA | 0.8176 |
| pyrene | 10,000.00 | 0.66 | 26.2 | 87.8 | 21.4 | 3.066 |
| benzo(a)anthracene* | 103.88 | 0.66 | NA | NA | NA | 0.01 |
| chrysene* | 10,000.00 | 0.66 | NA | NA | NA | 0.3918 |
| benzo(b)fluoanthene* | 354.98 | 0.66 | NA | NA | NA | 0.01 |
| benzo(k)fluoranthene* | 3,759.12 | 0.66 | NA | NA | NA | 0.0392 |
| benzo(a)pyrene | 69.85 | 0.66 | NA | NA | NA | 0.01 |
| indeno(1,2,3-cd)pyrene* | 629.17 | 0.66 | NA | NA | NA | 0.01 |
| dibenzo(a,h)anthracene* | 69.86 | 0.66 | NA | NA | NA | 0.01 |
| benzo(g,h,i)perylene | NA | 0.66 | NA | NA | NA | NA |
| 3,3'-dichlorobenzidine | 12.86 | 1.3 | NA | NA | NA | 0.02 |
| n-nitroso-di-n-propylamine | 0.66 | 0.66 | 30.7 | 90 | 34 | 0.01 |
| bis(2-chloroisopropyl)ether | 1.32 | 0.66 | NA | NA | NA | 0.0409 |
| 4-chloroaniline | 1,117.69 | 1.3 | NA | NA | NA | 0.4088 |
| 2-chloronaphthalene | 10,000.00 | 0.66 | NA | NA | NA | 8.176 |
| 2,4-dinitrotoluene | 39.07 | 0.66 | 28.6 | 80 | 12.5 | 0.2044 |
| hexachlorobutadiene | 31.18 | 0.66 | NA | NA | NA | 0.0367 |
| hexachloroethane | 3.31 | 0.66 | NA | NA | NA | 0.0204 |
| isophorone | 256.03 | 0.66 | NA | NA | NA | 3.0105 |
| benzyl alcohol | 4,356.75 | 1.3 | NA | NA | NA | 30.66 |
| bis(2-chloroethyl)ether | 0.66 | 0.66 | NA | NA | NA | 0.01 |
| nitrobenzene | 1.73 | 0.66 | NA | NA | NA | 0.0511 |
| 1,2-dichlorobenzene | 10,000.00 | 0.66 | NA | NA | NA | 9.198 |
| 1,3-dichlorobenzene | NA | 0.66 | NA | NA | NA | NA |
| 1,4-dichlorobenzene | 34.67 | 0.66 | 27.8 | 73.3 | 31.1 | 0.1192 |
| 1,2,4-trichlorobenzene | 1,405.37 | 0.66 | 26.4 | 74.6 | 34.6 | 1.022 |
| hexachlorobenzene | 101.56 | 0.66 | NA | NA | NA | 0.01 |
| hexachlorocyclopentadiene | 2.89 | 0.66 | NA | NA | NA | 0.7154 |
| n-nitrosodiphenylamine | 567.8 | 0.66 | NA | NA | NA | 0.5837 |
| benzoic acid | 10,000.00 | 3.3 | NA | NA | NA | 408.8 |
| 2-nitroaniline | 3.3 | 3.3 | NA | NA | NA | 0.05 |
| phenol | 658.78 | 0.66 | 28.1 | 77 | 31.4 | 12.264 |
| 2-methylphenol | 375.93 | 0.66 | NA | NA | NA | 5.11 |
| 3-methylphenol | NA | 0.66 | NA | NA | NA | NA |
| 4-methylphenol | 427.24 | 0.66 | NA | NA | NA | 5.11 |

[redacted]

| PQL(3) | Accuracy (5) | | Precision (5) |
|---|---|---|---|
| | LCL | UCL | RPD |
| (mg/l) | % | % | % |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | 28.6 | 88.6 | 41.3 |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | 25.3 | 103 | 37.7 |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.02 | NA | NA | NA |
| 0.01 | 15.9 | 119 | 42.7 |
| 0.01 | NA | NA | NA |
| 0.02 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | 23.5 | 95.4 | 36.5 |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.02 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | 20.6 | 82 | 39.7 |
| 0.01 | 19.7 | 80.9 | 44.8 |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.05 | NA | NA | NA |
| 0.05 | NA | NA | NA |
| 0.01 | 5 | 46.7 | 70.4 |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |

| | | | | | | |
|---|---|---|---|---|---|---|
| 2-chlorophenol | 11.63 | 0.66 | 29.4 | 75.7 | 32 | 0.511 |
| 2,4-dichlorophenol | 15.12 | 0.66 | NA | NA | NA | 0.3066 |
| 2,4,5-trichlorophenol | 5,507.44 | 0.66 | NA | NA | NA | 10.22 |
| 2,4,6-trichlorophenol | 30.65 | 0.66 | NA | NA | NA | 0.26 |
| pentachlorophenol | 24.95 | 3.3 | 5 | 106 | 12.2 | 0.05 |
| 2,4-dinitrophenol | 7.37 | 3.3 | NA | NA | NA | 0.2044 |
| bis(2-ethylhexyl)phthalate | 1,406.25 | 0.66 | NA | NA | NA | 0.2043 |
| butylbenzylphthalate | 10,000.00 | 0.66 | NA | NA | NA | 20.44 |
| di-n-butylphthalate | 6,188.56 | 0.66 | NA | NA | NA | 2.044 |
| diethylphthalate | 10,000.00 | 0.66 | NA | NA | NA | 81.76 |
| de methyl phthalate | 10,000.00 | 0.66 | NA | NA | NA | 1022 |
| di-n-octyl phthalate | 10,000.00 | 0.66 | NA | NA | NA | 2.044 |
| benzene | 4.77 | 0.005 | 85.8 | 120 | 32 | 0.0986 |
| toluene | 1,000.00 | 0.005 | 80.1 | 126 | 44.7 | 20.44 |
| ethylbenzene | 1,000.00 | 0.005 | 83.8 | 129 | 70.9 | 10.22 |
| xylenes | 1,000.00 | 0.005 | NA | NA | NA | 204.4 |
| vinyl chloride | 0.13 | 0.01 | 72.2 | 131 | 49.3 | 0.01 |
| chloroethane | 1,000.00 | 0.01 | 82.6 | 129 | 45 | NA |
| 1,1-dichloroethylene | 0.08 | 0.005 | 72.8 | 126 | 36.4 | 0.007 |
| 1,1-dichloroethane | 1,000.00 | 0.005 | 86.3 | 110 | 24.1 | 10.22 |
| 1,2-dichloroethylene (cis) | 102.49 | 0.005 | NA | NA | NA | 1.022 |
| 1,2-dichloroethane | 0.37 | 0.005 | 70.7 | 139 | 41.2 | 0.0314 |
| trichloroethylene | 25.73 | 0.005 | 82.6 | 116 | 35.3 | 0.26 |
| 1,1,1-trichloroethane | 1,000.00 | 0.005 | 86.2 | 124 | 24.4 | 9.198 |
| 1,1,2-trichloroethane | 1.05 | 0.005 | 72 | 137 | 34.4 | 0.0502 |
| tetrachloroethylene | 8.01 | 0.005 | 80 | 128 | 29.1 | 0.0561 |
| 1,1,1,2-tetrachloroethane | 7.24 | 0.005 | NA | NA | NA | 0.11 |
| 1,1,2,2-tetrachloroethane | 0.21 | 0.005 | 77.1 | 145 | 85.2 | 0.0143 |
| chloroform | 20.33 | 0.005 | 85.3 | 114 | 22.1 | 0.4689 |
| acetone | 136.29 | 0.1 | NA | NA | NA | 10.22 |
| 4-methyl-2-pentanone | 407.48 | 0.05 | NA | NA | NA | 5.11 |
| methyl ethyl ketone | 146.24 | 0.1 | NA | NA | NA | 5.11 |
| Aldrin | 0.06 | 0.003 | 50 | 150 | 30 | 0.0002 |
| gamma-BHC (Lindane) | 0.34 | 0.006 | 50 | 150 | 30 | 0.0022 |
| chlordane | 4.51 | 0.009 | NA | NA | NA | 0.002 |
| DDD | 48.34 | 0.007 | 50 | 150 | 30 | 0.0119 |
| DDE | 80.49 | 0.003 | 50 | 150 | 30 | 0.0084 |
| DDT | 141.83 | 0.008 | 50 | 150 | 30 | 0.0084 |
| dieldrin | 0.06 | 0.001 | 50 | 150 | 30 | 0.0002 |
| endosulfan sulfate | 12 | 0.044 | 50 | 150 | 30 | 0.0051 |
| endrin | 10.12 | 0.004 | 50 | 150 | 30 | 0.0061 |
| heptachlor | 0.44 | 0.002 | 50 | 150 | 30 | 0.0006 |
| heptachlor epoxide | 0.45 | 0.056 | 50 | 150 | 30 | 0.0008 |
| PCBs (Aroclor 1016) | 4.23 | 0.044 | 51.7 | 133 | 60.3 | 0.0007 |
| lead | NA | 0.5 | NA | NA | NA | NA |
| cadmium | 730 | 0.5 | NA | NA | NA | 0.0511 |
| silver | 7,300.00 | 1 | NA | NA | NA | 0.511 |
| mercury | 87.6 | 0.1 | NA | NA | NA | 0.0061 |
| chromium vi | 7,300.00 | 1 | 80 | 120 | 20 | 0.511 |
| chromium iii | 10,000.00 | 1 | NA | NA | NA | 102.2 |
| barium | 10,000.00 | 20 | NA | NA | NA | 7.154 |

| | | | | | | |
|---|---|---|---|---|---|---|
| arsenic | 438 | 1 | NA | NA | NA | 0.05 |
| antimony | 584 | 6 | NA | NA | NA | 0.06 |
| beryllium | 118.6 | 0.5 | NA | NA | NA | 0.005 |
| cyanide | 10,000.00 | 0.125 | NA | NA | NA | 2.044 |
| nickel | 10,000.00 | 4 | NA | NA | NA | 2.044 |
| selenium | 7,300.00 | 0.5 | NA | NA | NA | 0.511 |
| vanadium | 10,000.00 | 5 | NA | NA | NA | 0.7154 |
| zinc | 10,000.00 | 2 | NA | NA | NA | 30.66 |

Footnotes:

1. Data Quality Objectives for

2. Data Quality Objectives for

3. PQL - Practical Quantitation Limit, based on EPA SW-846, 1986 for GC/MS.
4. Subject to change based on current laboratory in-house acceptance limits.
5. Subject to change based on vendor supplied acceptance limits for a solid matrix standard.

NA- Not Available since organic

| | | | |
|---|---|---|---|
| 0.01 | 31.3 | 81.3 | 44.3 |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.05 | 5 | 114 | 29.2 |
| 0.05 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.01 | NA | NA | NA |
| 0.005 | 74.1 | 119 | 23.7 |
| 0.005 | 79.6 | 122 | 25.4 |
| 0.005 | 71.2 | 140 | 25.2 |
| 0.005 | NA | NA | NA |
| 0.01 | 37.2 | 152 | 31.2 |
| 0.01 | 5 | 347 | 29.3 |
| 0.005 | 56.6 | 124 | 31 |
| 0.005 | 64.3 | 124 | 14.2 |
| 0.005 | NA | NA | NA |
| 0.005 | 73.3 | 126 | 23.3 |
| 0.005 | 70.7 | 127 | 26.9 |
| 0.005 | 72.1 | 129 | 29 |
| 0.005 | 75.6 | 123 | 25.9 |
| 0.005 | 71.7 | 139 | 29.7 |
| 0.005 | NA | NA | NA |
| 0.005 | 54.8 | 144. | 27.3 |
| 0.005 | 75.8 | 116 | 25 |
| 0.1 | NA | NA | NA |
| 0.05 | NA | NA | NA |
| 0.1 | NA | NA | NA |
| 0.00004 | 50 | 150 | 30 |
| 0.00009 | 50 | 150 | 30 |
| 0.00014 | NA | NA | NA |
| 0.00011 | 50 | 150 | 30 |
| 0.00004 | 50 | 150 | 30 |
| 0.00012 | 50 | 150 | 30 |
| 0.00002 | 50 | 150 | 30 |
| 0.00066 | 50 | 150 | 30 |
| 0.00006 | 50 | 150 | 30 |
| 0.00003 | 50 | 150 | 30 |
| 0.00083 | 50 | 150 | 30 |
| 0.00065 | 30.7 | 153 | 30 |
| 0.003 | 80 | 120 | 20 |
| 0.005 | 80 | 120 | 20 |
| 0.01 | 85 | 115 | 20 |
| 0.0002 | 85 | 115 | 20 |
| 0.01 | 90 | 110 | 20 |
| 0.01 | NA | NA | NA |
| 0.2 | 85 | 115 | 20 |

| | | | |
|---|---|---|---|
| 0.01 | 80 | 120 | 20 |
| 0.06 | 80 | 120 | 20 |
| 0.005 | 88 | 115 | 20 |
| 0.01 | 90 | 110 | 20 |
| 0.04 | 85 | 115 | 20 |
| 0.005 | 80 | 120 | 20 |
| 0.05 | 85 | 115 | 20 |
| 0.02 | 85 | 115 | 20 |

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil [1] Nonresidential | | | | | Groundwater [2] Nonresidential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level (mg/kg) | PQL [3] (mg/kg) | Accuracy [4] LCL % | Accuracy [4] UCL % | Precision [4] RPD % | Screening Level (mg/l) | PQL [3] (mg/l) | Accuracy [5] LCL % | Accuracy [5] UCL % | Precision [5] RPD % |
| naphthalene | 10,000.00 | 0.66 | NA | NA | NA | 4.088 | 0.01 | NA | NA | NA |
| acenaphthylene | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| acenaphthene | 10,000.00 | 0.66 | 31 | 78.8 | 24.5 | 6.132 | 0.01 | 28.6 | 88.6 | 41.3 |
| fluorene | 10,000.00 | 0.66 | NA | NA | NA | 4.088 | 0.01 | NA | NA | NA |
| phenanthrene | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| anthracene | 10,000.00 | 0.66 | NA | NA | NA | 30.66 | 0.01 | NA | NA | NA |
| fluoranthene | 10,000.00 | 0.66 | NA | NA | NA | 0.8176 | 0.01 | NA | NA | NA |
| pyrene | 10,000.00 | 0.66 | 26.2 | 87.8 | 21.4 | 3.066 | 0.01 | 25.3 | 103 | 37.7 |
| benzo(a)anthracene* | 103.88 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| chrysene* | 10,000.00 | 0.66 | NA | NA | NA | 0.3918 | 0.01 | NA | NA | NA |
| benzo(b)fluoanthene* | 354.98 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| benzo(k)fluoranthene* | 3,759.12 | 0.66 | NA | NA | NA | 0.0392 | 0.01 | NA | NA | NA |
| benzo(a)pyrene | 69.85 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| indeno(1,2,3-cd)pyrene* | 629.17 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| dibenzo(a,h)anthracene* | 69.86 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| benzo(g,h,i)perylene | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| 3,3'-dichlorobenzidine | 12.86 | 1.3 | NA | NA | NA | 0.02 | 0.02 | NA | NA | NA |
| n-nitroso-di-n-propylamine | 0.66 | 0.66 | 30.7 | 90 | 34 | 0.01 | 0.01 | 15.9 | 119 | 42.7 |
| bis(2-chloroisopropyl)ether | 1.32 | 0.66 | NA | NA | NA | 0.0409 | 0.01 | NA | NA | NA |
| 4-chloroaniline | 1,117.69 | 1.3 | NA | NA | NA | 0.4088 | 0.02 | NA | NA | NA |
| 2-chloronaphthalene | 10,000.00 | 0.66 | NA | NA | NA | 8.176 | 0.01 | NA | NA | NA |
| 2,4-dinitrotoluene | 39.07 | 0.66 | 28.6 | 80 | 12.5 | 0.2044 | 0.01 | 23.5 | 95.4 | 36.5 |
| hexachlorobutadiene | 31.18 | 0.66 | NA | NA | NA | 0.0367 | 0.01 | NA | NA | NA |
| hexachloroethane | 3.31 | 0.66 | NA | NA | NA | 0.0204 | 0.01 | NA | NA | NA |
| isophorone | 256.03 | 0.66 | NA | NA | NA | 3.0105 | 0.01 | NA | NA | NA |
| benzyl alcohol | 4,356.75 | 1.3 | NA | NA | NA | 30.66 | 0.02 | NA | NA | NA |
| bis(2-chloroethyl)ether | 0.66 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| nitrobenzene | 1.73 | 0.66 | NA | NA | NA | 0.0511 | 0.01 | NA | NA | NA |
| 1,2-dichlorobenzene | 10,000.00 | 0.66 | NA | NA | NA | 9.198 | 0.01 | NA | NA | NA |
| 1,3-diehlorobenzene | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| 1,4-dichlorobenzene | 34.67 | 0.66 | 27.8 | 73.3 | 31.1 | 0.1192 | 0.01 | 20.6 | 82 | 39.7 |
| 1,2,4-trichlorobenzene | 1,405.37 | 0.66 | 26.4 | 74.6 | 34.6 | 1.022 | 0.01 | 19.7 | 80.9 | 44.8 |
| hexachlorobenzene | 101.56 | 0.66 | NA | NA | NA | 0.01 | 0.01 | NA | NA | NA |
| hexachlorocyclopentadiene | 2.89 | 0.66 | NA | NA | NA | 0.7154 | 0.01 | NA | NA | NA |
| n-nitrosodiphenylamine | 567.8 | 0.66 | NA | NA | NA | 0.5837 | 0.01 | NA | NA | NA |
| benzoic acid | 10,000.00 | 3.3 | NA | NA | NA | 408.8 | 0.05 | NA | NA | NA |
| 2-nitroaniline | 3.3 | 3.3 | NA | NA | NA | 0.05 | 0.05 | NA | NA | NA |
| phenol | 658.78 | 0.66 | 28.1 | 77 | 31.4 | 12.264 | 0.01 | 5 | 46.7 | 70.4 |
| 2-methylphenol | 375.93 | 0.66 | NA | NA | NA | 5.11 | 0.01 | NA | NA | NA |
| 3-methylphenol | NA | 0.66 | NA | NA | NA | NA | 0.01 | NA | NA | NA |
| 4-methylphenol | 427.24 | 0.66 | NA | NA | NA | 5.11 | 0.01 | NA | NA | NA |
| 2-chlorophenol | 11.63 | 0.66 | 29.4 | 75.7 | 32 | 0.511 | 0.01 | 31.3 | 81.3 | 44.3 |
| 2,4-dichlorophenol | 15.12 | 0.66 | NA | NA | NA | 0.3066 | 0.01 | NA | NA | NA |
| 2,4,5-trichlorophenol | 5,507.44 | 0.66 | NA | NA | NA | 10.22 | 0.01 | NA | NA | NA |
| 2,4,6-trichlorophenol | 30.65 | 0.66 | NA | NA | NA | 0.26 | 0.01 | NA | NA | NA |
| pentachlorophenol | 24.95 | 3.3 | 5 | 106 | 12.2 | 0.05 | 0.05 | 5 | 114 | 29.2 |
| 2,4-dinitrophenol | 7.37 | 3.3 | NA | NA | NA | 0.2044 | 0.05 | NA | NA | NA |
| bis(2-ethylhexyl)phthalate | 1,406.25 | 0.66 | NA | NA | NA | 0.2043 | 0.01 | NA | NA | NA |
| butylbenzylphthalate | 10,000.00 | 0.66 | NA | NA | NA | 20.44 | 0.01 | NA | NA | NA |
| di-n-butylphthalate | 6,188.56 | 0.66 | NA | NA | NA | 2.044 | 0.01 | NA | NA | NA |
| diethylphthalate | 10,000.00 | 0.66 | NA | NA | NA | 81.76 | 0.01 | NA | NA | NA |
| de methyl phthalate | 10,000.00 | 0.66 | NA | NA | NA | 1022 | 0.01 | NA | NA | NA |
| di-n-octyl phthalate | 10,000.00 | 0.66 | NA | NA | NA | 2.044 | 0.01 | NA | NA | NA |
| benzene | 4.77 | 0.005 | 85.8 | 120 | 32 | 0.0986 | 0.005 | 74.1 | 119 | 23.7 |
| toluene | 1,000.00 | 0.005 | 80.1 | 126 | 44.7 | 20.44 | 0.005 | 79.6 | 122 | 25.4 |

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Cnnstituent | Subsurface Snil [1] Nonresidential | | | | | Grouudwater[2] Nonresidential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level (mg/kg) | PQL[3] (mg/kg) | Accuracy [4] LCL % | Accuracy [4] UCL % | Precision [4] RPD % | Screening Level (mg/l) | PQL[3] (mg/l) | Accuracy [5] LCL % | Accuracy [5] UCL % | Precision [5] RPD % |
| ethylbenzene | 1,000.00 | 0.005 | 83.8 | 129 | 70.9 | 10.22 | 0.005 | 71.2 | 140 | 25.2 |
| xylenes | 1,000.00 | 0.005 | NA | NA | NA | 204.4 | 0.005 | NA | NA | NA |
| vinyl chloride | 0.13 | 0.01 | 72.2 | 131 | 49.3 | 0.01 | 0.01 | 37.2 | 152 | 31.2 |
| chloroethane | 1,000.00 | 0.01 | 82.6 | 129 | 45 | NA | 0.01 | 5 | 347 | 29.3 |
| 1,1-dichloroethylene | 0.08 | 0.005 | 72.8 | 126 | 36.4 | 0.007 | 0.005 | 56.6 | 124 | 31 |
| 1,1-dichloroethane | 1,000.00 | 0.005 | 86.3 | 110 | 24.1 | 10.22 | 0.005 | 64.3 | 124 | 14.2 |
| 1,2-dichloroethylene (cis) | 102.49 | 0.005 | NA | NA | NA | 1.022 | 0.005 | NA | NA | NA |
| 1,2-dichloroethane | 0.37 | 0.005 | 70.7 | 139 | 41.2 | 0.0314 | 0.005 | 73.3 | 126 | 23.3 |
| trichloroethylene | 25.73 | 0.005 | 82.6 | 116 | 35.3 | 0.26 | 0.005 | 70.7 | 127 | 26.9 |
| 1,1,1-trichloroethane | 1,000.00 | 0.005 | 86.2 | 124 | 24.4 | 9.198 | 0.005 | 72.1 | 129 | 29 |
| 1,1,2-trichloroethane | 1.05 | 0.005 | 72 | 137 | 34.4 | 0.0502 | 0.005 | 75.6 | 123 | 25.9 |
| tetrachloroethylene | 8.01 | 0.005 | 80 | 128 | 29.1 | 0.0561 | 0.005 | 71.7 | 139 | 29.7 |
| 1,1,1,2-tetrachloroethane | 7.24 | 0.005 | NA | NA | NA | 0.11 | 0.005 | NA | NA | NA |
| 1,1,2,2-tetrachloroethane | 0.21 | 0.005 | 77.1 | 145 | 85.2 | 0.0143 | 0.005 | 54.8 | 144 | 27.3 |
| chloroform | 20.33 | 0.005 | 85.3 | 114 | 22.1 | 0.4689 | 0.005 | 75.8 | 116 | 25 |
| acetone | 136.29 | 0.1 | NA | NA | NA | 10.22 | 0.1 | NA | NA | NA |
| 4-methyl-2-pentanone | 407.48 | 0.05 | NA | NA | NA | 5.11 | 0.05 | NA | NA | NA |
| methyl ethyl ketone | 146.24 | 0.1 | NA | NA | NA | 5.11 | 0.1 | NA | NA | NA |
| Aldrin | 0.06 | 0.003 | 50 | 150 | 30 | 0.0002 | 0.00004 | 50 | 150 | 30 |
| gamma-BHC (Lindane) | 0.34 | 0.006 | 50 | 150 | 30 | 0.0022 | 0.00009 | 50 | 150 | 30 |
| chlordane | 4.51 | 0.009 | NA | NA | NA | 0.002 | 0.00014 | NA | NA | NA |
| DDD | 48.34 | 0.007 | 50 | 150 | 30 | 0.0119 | 0.00011 | 50 | 150 | 30 |
| DDE | 80.49 | 0.003 | 50 | 150 | 30 | 0.0084 | 0.00004 | 50 | 150 | 30 |
| DDT | 141.83 | 0.008 | 50 | 150 | 30 | 0.0084 | 0.00012 | 50 | 150 | 30 |
| dieldrin | 0.06 | 0.001 | 50 | 150 | 30 | 0.0002 | 0.00002 | 50 | 150 | 30 |
| endosulfan sulfate | 12 | 0.044 | 50 | 150 | 30 | 0.0051 | 0.00066 | 50 | 150 | 30 |
| endrin | 10.12 | 0.004 | 50 | 150 | 30 | 0.0061 | 0.00006 | 50 | 150 | 30 |
| heptachlor | 0.44 | 0.002 | 50 | 150 | 30 | 0.0006 | 0.00003 | 50 | 150 | 30 |
| heptachlor epoxide | 0.45 | 0.056 | 50 | 150 | 30 | 0.0008 | 0.00083 | 50 | 150 | 30 |
| PCBs (Aroclor 1016) | 4.23 | 0.044 | 51.7 | 133 | 60.3 | 0.0007 | 0.00065 | 30.7 | 153 | 30 |
| lead | NA | 0.5 | NA | NA | NA | NA | 0.003 | 80 | 120 | 20 |
| cadmium | 730 | 0.5 | NA | NA | NA | 0.0511 | 0.005 | 80 | 120 | 20 |
| silver | 7,300.00 | 1 | NA | NA | NA | 0.511 | 0.01 | 85 | 115 | 20 |
| mercury | 87.6 | 0.1 | NA | NA | NA | 0.0061 | 0.0002 | 85 | 115 | 20 |
| chromium vi | 7,300.00 | 1 | 80 | 120 | 20 | 0.511 | 0.01 | 90 | 110 | 20 |
| chromium iii | 10,000.00 | 1 | NA | NA | NA | 102.2 | 0.01 | NA | NA | NA |
| barium | 10,000.00 | 20 | NA | NA | NA | 7.154 | 0.2 | 85 | 115 | 20 |
| arsenic | 438 | 1 | NA | NA | NA | 0.05 | 0.01 | 80 | 120 | 20 |
| antimony | 584 | 6 | NA | NA | NA | 0.06 | 0.06 | 80 | 120 | 20 |
| beryllium | 118.6 | 0.5 | NA | NA | NA | 0.005 | 0.005 | 88 | 115 | 20 |
| cyanide | 10,000.00 | 0.125 | NA | NA | NA | 2.044 | 0.01 | 90 | 110 | 20 |
| nickel | 10,000.00 | 4 | NA | NA | NA | 2.044 | 0.04 | 85 | 115 | 20 |
| selenium | 7,300.00 | 0.5 | NA | NA | NA | 0.511 | 0.005 | 80 | 120 | 20 |
| vanadium | 10,000.00 | 5 | NA | NA | NA | 0.7154 | 0.05 | 85 | 115 | 20 |
| zinc | 10,000.00 | 2 | NA | NA | NA | 30.66 | 0.02 | 85 | 115 | 20 |

Footnotes:

1. Data Quality Objectives for subsurface soil are taken from Indian Department of Environmetal Management, Summary of Health-Based Criteria for Subsurface Soils (Nonresidential Land-Use Scenario), in the Resource Guide for the Indiana Voluntary Remediation Program, Appendix F, Table 10 (July 1996).
2. Data Quality Objectives for subsurface soil are taken from Indian Department of Environmetal Management, Summary of Health-Based Criteria for Subsurface Soils (Nonresidential Land-Use Scenario), in the Resource Guide for the Indiana Voluntary Remediation Program, Appendix F, Table 10 (July 1996).
3. PQL - Practical Quantitation Limit, based on EPA SW-846, 1986 for GC/MS.
4. Subject to change based on current laboratory in-house acceptance limits.
5. Subject to change based on vendor supplied acceptance limits for a solid matrix standard.

NA- Not Applicable since organic cnnstituents are spiked and controlled. List of spiked constituents are defined in method, however, the spike list is subject to change.

Jan

The following is in response to your last e-mail. In addition I, have attached an updated version of table 1-2. If you have any question, please let me know.

Thanks,
Rick Spitaler

1 Regarding the analyses for the metals, I'm still not clear about what
list of metals you'll be looking for. The sampling plan (Table 3-2) identifies the 8 RCRA metals (As, Ba, Cd, Cr, Pb, Hg, Se and Ag); however, the reply below includes some additional metals (such as Sb, Ni, V and Zn). The metals list needs to be resolved and corrected such that it's clear what will be analyzed for.

The sampling plan is correct in indicating only the 8 RCRA metals. The lab inadvertently included Sb, Ni, V and Zn in their response and I ended up passing on the information. These metals should not have been included.

2 Based on the reply below, we are still missing the 1) lab SOPs for the
GFAA sample prep for water water samples, and the 2) lab SOP for GFAA of Cd in waters.

The following SOPs requested are Attached for your review:

Standard operating procedures for the preparation of aqueous samples and extracts for total or dissolved metals analysis by Graphite Furnace Atomic Absorption Spectroscopy

Standard operating procedures for atomic absorption analysis of cadmium for aqueous samples

3 Table 3-1 of the sampling plan needs to reflect that the list of PAHs
below will be analyzed for by Method 8270 in soils, and by Method 8310
for waters.

Table 3-1 has been updated and is included as an attachment

4 You may want to itemize the list of analytes that are required and
attach this list to the sampling plan , similar to what you have below.
You may want to do the same for the VOC compounds, so that it's clear whether you're going to look for the entire list of VOCs in Table 1-2 or
some sub-set.

Attached is a list of analytes provided by Sima

5 Does the lab have a list of soil MDLs for the metals?

Sima Labs provided the following response to your request.

Here is the MDL data. The MDLs are performed in water (thereby explaining the mg/l units). A "typical" preparation factor for soil

samples is 100 (1g sample digested and diluted to a final volume of 100ml). The aqueous MDL is multiplied by this preparation factor to estimate the MDL for a solid matrix. Please realize that SIMALABS International does not encourage reporting data down to the MDL, as the
probability for false biased data is considered too great.

| ELEMENT | AQUEOUS MDL, mg/l | SOLID MDL, mg/kg |
|---|---|---|
| As (ICP) | 0.035 | 3.5 |
| As (GFAA) | 0.005 | 0.5 |
| Ba (ICP) | 0.001 | 0.1 |
| Cd (ICP) | 0.002 | 0.2 |
| Cd (GFAA) | 0.00003 | 0.003 |
| Cr (ICP) | 0.003 | 0.3 |
| Pb (ICP) | 0.0025 | 0.2 |
| Pb (GFAA) | 0.002 | 0.2 |
| Se (ICP) | 0.036 | 3.6 |
| Se (GFAA) | 0.002 | 0.2 |
| Ag (ICP) | 0.003 | 0.3 |
| Hg (CVAA) | 0.00003 | 0.003 |

8260_8270Cmpds-PQLs.doTable 1-2 Final.xlssamples 3-1.xls

| | |
|---|---|
| 1,2-Dibromoethane | 5 |
| 1,2-Dichlorobenzene | 10 |
| 1,3,5-Trimethylbenzene | 5 |
| 1,3-Dichlorobenzene | 10 |
| 1,3-Dichloropropane | 5 |
| 1,4-Dichlorobenzene | 10 |
| 2,2-Dichloropropane | 5 |
| 2-Chloroethyl vinyl ether | 10 |
| 2-Chlorotoluene | 10 |
| 4-Chlorotoluene | 10 |
| Acetonitrile | 100 |
| Bromobenzene | 5 |
| Cumene | 5 |
| Dibromomethane | 5 |
| Dichlorodifluoromethane | 5 |
| Ethylene Dibromide | 10 |
| Hexachlorobutadiene | 100 |
| Isopropylbenzene | 5 |
| n-Butyl Alcohol | 100 |
| n-Butylbenzene | 10 |
| n-Propylbenzene | 10 |
| Naphthalene | 10 |
| p-Isopropyltoluene | 5 |
| sec-Butylbenzene | 5 |
| tert-Butylbenzene | 5 |
| Total 1,2-Dichloroethene | 5 |
| Total Xylenes | 5 |

| SVOCs by 8270C |
|---|

| VOCs by 8260B | |
|---|---|
| **Analyte** | **PQL, ug/l** |
| 1,1,1,2-Tetrachloroethane | 10 |
| 1,1,1-Trichloroethane | 5 |
| 1,1,2,2-Tetrachloroethane | 5 |
| 1,1,2-Trichloroethane | 5 |
| 1,1-Dichloroethane | 5 |
| 1,1-Dichloroethene | 5 |
| 1,2-Dichloroethane | 5 |
| 1,2-Dichloropropane | 5 |
| 2-Butanone | 10 |
| 2-Hexanone | 10 |
| 4-Methyl-2-Pentanone | 10 |
| Acetone | 50 |
| Acrolein | 100 |
| Acrylonitrile | 100 |
| Benzene | 5 |
| Bromodichloromethane | 5 |
| Bromoform | 5 |
| Bromomethane | 10 |
| Carbon Disulfide | 10 |
| Carbon tetrachloride | 5 |
| Chlorobenzene | 5 |
| Chlorodibromomethane | 5 |
| Chloroethane | 10 |
| Chloroform | 5 |
| Chloromethane | 10 |
| cis-1,2-Dichloroethene | 5 |
| cis-1,3-Dichloropropene | 5 |
| Dibromochloromethane | 5 |
| Dichlorobromomethane | 5 |
| Dichloromethane | 5 |
| Ethylbenzene | 5 |
| m,p-Xylene | 5 |
| Methyl Ethyl Ketone | 10 |
| Methyl Isobutyl Ketone | 5 |
| Methyl-t-Butyl Ether | 10 |
| Methylene chloride | 10 |
| MTBE | 10 |
| o-Xylene | 5 |
| Styrene | 5 |
| tert-Butyl Methyl Ether | 10 |
| Tetrachloroethene | 5 |
| Toluene | 5 |
| trans-1,2-Dichloroethene | 5 |
| trans-1,3-Dichloropropene | 5 |
| Trichloroethene | 5 |
| Trichlorofluoromethane | 5 |
| Vinyl Acetate | 10 |
| Vinyl chloride | 10 |
| 1,1-Dichloropropene | 5 |
| 1,2,3-Trichlorobenzene | 5 |
| 1,2,3-Trichloropropane | 5 |
| 1,2,4-Trichlorobenzene | 5 |
| 1,2,4-Trimethylbenzene | 5 |
| 1,2-Dibromo-3-Chloropropane | 5 |

| Analyte | PQL, ug/l |
|---|---|
| 1,2,4-Trichlorobenzene | 10 |
| 1,2-Dichlorobenzene | 10 |
| 1,2-Diphenyl-hydrazine | 10 |
| 1,3-Dichlorobenzene | 10 |
| 1,4-Dichlorobenzene | 10 |
| 2,2'-oxybis(1-chloropropane) | 10 |
| 2,4,5-Trichlorophenol | 10 |
| 2,4,6-Trichlorophenol | 10 |
| 2,4-Dichlorophenol | 10 |
| 2,4-Dimethylphenol | 10 |
| 2,4-Dinitrophenol | 50 |
| 2,4-Dinitrotoluene | 10 |
| 2,6-Dichlorophenol | 10 |
| 2,6-Dinitrotoluene | 10 |
| 2-Chloronaphthalene | 10 |
| 2-Chlorophenol | 10 |
| 2-Methyl-4,6-dinitrophenol | 50 |
| 2-Methylnaphthalene | 10 |
| 2-Methylphenol | 10 |
| 2-Nitroaniline | 50 |
| 2-Nitrophenol | 10 |
| 3,3'-Dichlorobenzidine | 50 |
| 3,4-Benzofluoranthene | 10 |
| 3-Methylphenol | 10 |
| 3-Nitroaniline | 50 |
| 3/4-Methylphenol | 10 |
| 4,6-Dinitro-2-methylphenol | 50 |
| 4,6-Dinitro-o-cresol | 50 |
| 4-Bromophenyl phenyl ether | 10 |
| 4-Chloro-3-methylphenol | 20 |
| 4-Chloroaniline | 20 |
| 4-Chlorophenyl phenyl ether | 10 |
| 4-Nitroaniline | 50 |
| 4-Nitrophenol | 50 |
| Acenaphthene | 10 |
| Acenaphthylene | 10 |
| Acetophenone | 10 |
| Aniline | 10 |
| Anthracene | 10 |
| Benzidine | 50 |
| Benzo[a]anthracene | 10 |
| Benzo[a]pyrene | 10 |
| Benzo[b]fluoranthene | 10 |
| Benzo[g,h,i]perylene | 10 |
| Benzo[j]fluoranthene | 10 |
| Benzo[k]fluoranthene | 10 |
| Benzoic acid | 50 |
| Benzyl alcohol | 20 |
| beta-Chloronaphthalene | 10 |
| Bis(2-chloroethoxy)methane | 10 |
| Bis(2-chloroethyl)ether | 10 |
| Bis(2-chloroisopropyl)ether | 10 |
| Bis(2-ethylhexyl)phthalate | 10 |
| Butyl benzyl phthalate | 10 |
| Carbazole | 10 |
| Chrysene | 10 |

| | |
|---|---|
| Di(2-ethylhexyl) phthalate | 10 |
| Di-n-butyl phthalate | 10 |
| Di-n-octyl phthalate | 10 |
| Dibenz[a,h]anthracene | 10 |
| Dibenzofuran | 10 |
| Diethyl phthalate | 10 |
| Dimethyl phthalate | 10 |
| Fluoranthene | 10 |
| Fluorene | 10 |
| Hexachlorobenzene | 10 |
| Hexachlorobutadiene | 10 |
| Hexachlorocyclopentadiene | 10 |
| Hexachloroethane | 10 |
| Indeno[1,2,3cd]pyrene | 10 |
| Isophorone | 10 |
| m-Dichlorobenzene | 10 |
| N-Nitrosodi-n-propylamine | 10 |
| N-Nitrosodimethylamine | 10 |
| N-Nitrosodiphenylamine | 10 |
| Naphthalene | 10 |
| Nitrobenzene | 10 |
| o-Chlorophenol | 10 |
| p-Chloro-m-cresol | 20 |
| p-Chloroaniline | 20 |
| p-Cresol | 10 |
| Pentachlorophenol | 50 |
| Phenanthrene | 10 |
| Phenol | 10 |
| Pyrene | 10 |
| Pyridine | 10 |
| Total Cresol | 10 |

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# STANDARD OPERATING PROCEDURE FOR HEXAVALENT CHROMIUM BY SM METHOD 3500-CR D AND SW-846 METHOD 7196A

Originating Author: Karin Stewart
Revision Author: Jeff Loewe

This SOP is effective upon signed approval by the following:

Unit Supervisor — Date: 6-14-2001

QA/QC Director — Date: 6-14-2001

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

## 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This is an automated colorimetric procedure for the determination of Hexavalent Chromium. This procedure is applicable to the direct analysis of aqueous samples and the digestates of solid matrix samples. The routine reporting limits are 0.005 mg/l and 16 mg/kg.

## 3.0 SUMMARY

3.1 Using an acidic solution of diphenylcarbazide, hexavalent chromium generates a red-violet color with maximum absorbance at 540 nm.

3.2 The linear working range is 0.005 to 0.400 mg/l.

## 4.0 DEFINITIONS

4.1 Accuracy – The degree of agreement of a measured value with the true or expected value of the quantity of concern (% recovery of a known spiked analyte).

4.2 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.3 Analyte – The specific component measured in a chemical analysis.

4.4 Analytical Batch – A group of samples which are analyzed, at the instrument level, together using the same method, reagents and apparatus within the same time period. Typically, these are samples in the same batch ID in the LIMS.

4.5 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Calibration Blank – An aliquot of the standard diluent (water or organic solvent) that is not carried through the sample preparation scheme. It is analyzed to verify that the analytical system is free from contamination. Also referred to as an instrument blank or solvent blank.

- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.

- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Method Blank.

4.6 Bias – The deviation of a measured value from a known or accepted value due to matrix effects or method performance. Bias may be determined quantitatively to correct measured values. Bias may be positive or negative.

4.7 Calibration – The establishment of an analytical curve based on the absorbance, response, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type and concentration of acids, solvents, or other solutions used in the sample preparation.

4.8 Continuing Calibration Verification Standard (CCV) – A standard used to verify the continued acceptability of the initial calibration curve. A continuing calibration verification must be repeated at the beginning and end of each analytical batch and every 10-20 samples, whichever is more frequent depending on the method requirements. The concentrations of the continuing calibration verification standard shall be varied within the established calibration range. If an internal standard is used, only one continuing calibration verification must be analyzed per analytical batch.

4.9 Detection Limit – The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

- IDL – Instrument detection limit. A statistically determined detection limit used to estimate the instrument's sensitivity. The IDL is obtained by analyzing seven consecutive blanks to assess the variability of the instrument.

- MDL – Method detection limit. The minimum concentration of a substance that can be measured and reported with a 99% degree of confidence. MDLs are determined by analyzing a minimum of seven consecutive standards that have been processed through all preparatory steps.

- PQL – The Practical Quantitation Limit is the lowest concentration that can reliably be achieved within specified limits of precision and accuracy during routine laboratory operating conditions. Typically, the PQL is a value in the range of 5 - 10 times the MDL. Also referred to as the Estimated Quantitation Limit (EQL).

4.10 Initial Calibration Verification (ICV) – A standard used to verify the accuracy of calibration standards. Prepared from a second source than that of the calibration standards, its known value is measured against the calibration curve. This determines the integrity of working standards. Also referred to as an external verification standard or check standard.

4.11 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.12 Laboratory Control Sample (LCS) – An aliquot of laboratory pure reagent spiked with target analytes or compounds representative of target analytes. The sample is

carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.13 Laboratory Control Sample Duplicate (LCSD) – An aliquot of laboratory pure reagent spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.14 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.15 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.16 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.17 Percent Recovery – A measure of accuracy that is calculated as the measured value relative to the true value, expressed as a percent.

$$\%R = \frac{MV}{TV} * 100$$

where: MV = measured value
TV = true value

4.18 Precision – The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the comparability of results from duplicate or replicate analyses. (%RPD between the recoveries of two known analyte spikes, and %RSD between the recoveries of three or more measurements).

4.19 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.20 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.21 Relative Percent Difference (% RPD) – Used to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

$$\% \text{ RPD} = \frac{|X - Y|}{(X + Y) / 2} * 100$$

where: X = value 1
Y = value 2

4.22 Sample – A portion of material supplied by the client for analysis.

## 5.0 INTERFERENCES

5.1 Substances that can reduce Cr (VI) upon acidification (cyanides, thiosulfate, organic matter) will cause negative interferences in the determination of Cr (VI).

5.2 In order to correct for sample color interference, any samples that are determined by this method to contain Cr (VI) are analyzed again but without the addition of the color reagent. This "color blank" value is subtracted from the original analytical result to account for indigenous sample color.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Analytical balance

7.3 Glass microfiber filters

7.4 1000 ml, 500 ml, and 250 ml volumetric flasks

7.5 Oxford 1-5 and 5-10 ml autopipettes

7.6 Lachat QuikChem 8000 FIA instrument including sampler, multichannel proportioning pump, reaction manifold, colorimetric detector, and data system.

7.7 13 x 100 mm disposable borosilicate glass culture tubes

7.8 Magnetic stirrer and stir bars

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook.

8.2 Reagents

8.2.1 Lab pure water

8.2.2 Sulfuric acid, conc. $H_2SO_4$

8.2.3 Isopropanol

8.2.4 Color Reagent: in a 1L volumetric flask, dissolve 0.40 g s-diphenylcarbazide in 200 ml isopropanol. Stir with magnetic stirrer until dissolved. Then add 720 ml water and 80.0 ml conc. sulfuric acid. Dilute to mark with water and mix with a magnetic stirrer.

8.3 Standards

8.3.1 Stock Calibration Standard, 1000 mg/l: Obtain from vendor.

8.3.2 Intermediate Calibration Standard, 20 mg/l: In a 250 ml volumetric flask, dilute 5.0 ml of the stock calibration standard to the mark with DI water.

8.3.3 Working Calibration Standard, 0.400 mg/l: In a 500 ml volumetric flask, dilute 10.0 ml of the intermediate calibration standard to the mark with DI water.

8.3.4 Prepare the calibration curve using the 0.400 mg/l standard and the following standards that are made from the 0.400 mg/l.

| ml. of 0.400 mg/l Std. | Final Vol., ml | Final Conc., mg/l |
|---|---|---|
| 50 | 100 | 0.200 |
| 25 | | 0.100 |
| 12.5 | | 0.050 |
| 2.5 | | 0.010 |
| 1.25 | | 0.005 |
| 0 | | 0 |

8.3.5 Stock Verification Standard, 1000 mg/l: This standard must be of a separate source or lot number from that used for calibration.

8.3.6 Intermediate Verification Standard, 20 mg/l: In a 250 ml volumetric flask, dilute 5.0 ml of the stock verification standard to the mark with DI water.

8.3.7 Working Verification Standard, 0.200 mg/l: In a 500 ml volumetric flask, dilute 5.0 ml of the intermediate verification standard to the mark with DI water.

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Samples should be collected in a plastic container. Preservation consists of storage in the range of 0.1-6°C.

9.3 Analysis must be performed within the maximum allowable hold time of 24 hours from collection.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Analyze four separate standards prepared in the range of 8-10 times the method detection limit listed in section 14.0. These standards must be from a source different from that used for calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation.

10.2 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Analyze a minimum of seven (maximum of ten) standards prepared in the range of 2-5 times the method detection limit listed in section 14.0 or an estimated detection limit. These standards must be taken through the entire analytical procedure. Submit the data to the QA department for evaluation.

10.3 A *Calibration Verification Standard* must be analyzed immediately after calibration, after every 10 samples, and after the sample. Acceptance criteria are 90.0 – 110% recovery. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier. Samples associated with a verification that fails with positive bias can be reported if the sample concentration is a non-detect.

10.4 A *Calibration Verification Blank* sample must be analyzed after each calibration verification standard. The acceptance criteria are < PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. If the blank does not meet the acceptance criteria, all samples < PQL or greater than 10 times the blank contamination may be reported. All other environmental samples must be reanalyzed or reported with an appropriate qualifier.

10.5 A *Method Blank* must be analyzed with each batch of maximum 20 digested samples and at a minimum of one per day digested. Acceptance criteria are < PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. If the blank does not meet the acceptance criteria, all samples < PQL or greater than 10 times the blank contamination may be reported. All other environmental samples must be reanalyzed or reported with an appropriate qualifier.

10.6 A *Laboratory Control Sample* must be analyzed with each batch of maximum 20 digested samples and at a minimum of one per day digested. Acceptance criteria are 80.0 – 120% recovery. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria all environmental samples must be reprepared and analyzed or reported with an appropriate qualifier. If the LCS fails with high bias, samples having a non-detectable concentration may be reported without qualification.

10.7 A *Matrix Spike and Matrix Spike Duplicate* sample must be analyzed with each batch of maximum 20 samples and at a minimum of one per day. Acceptance criteria for the MS are 85.0 – 115% (waters) and 75.0 – 125% (solids) recovery for accuracy, and $\leq$ 20.0% RPD for precision. If the acceptance criteria for either, accuracy or precision, are not met, reanalyze. If reanalysis fails to meet the acceptance criteria the sample and its MS/MSD must be reprepared and analyzed or reported with an appropriate qualifier.

10.8 Applicable to digested samples only, a *Post Digestion Spike* sample must be analyzed with each MS/MSD that do no meet the accuracy recovery criteria. Acceptance criteria are 85.0 – 115%. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria the sample and its MS/MSD must be reprepared and analyzed or reported with an appropriate qualifier.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Perform the required preventative maintenance as necessary.

11.2 Install the Lachat Cr (VI) manifold on the instrument in the Channel 2 position. Insert the 540 nm interference filter into detector module and connect sample loop (150 cm) in injection valve at the 1 and 4 positions. Detach pump tubing at the pump tube adaptor on the carrier line. Insert the tubing that is still attached to the manifold into the number 3 position on the injection valve and then attach the tubing from the number 2 position on the valve to remaining carrier line. Attach reagent and sample lines to pump with cassettes and switch on power to the instrument.

11.3 Load autosampler tray with standards and samples.

11.4 Log into Omnion. Open the Cr (VI) method and the tray.

11.5 Place reagent lines into DI water and pump through manifold until analysis is ready to start at which time the lines are placed into appropriate reagent containers.

11.6 Check for leaks and smooth flow.

11.7 Place the working calibration standard on the autosampler. Calibrate from high to low concentration.

11.8 Place reagent transmission lines into the appropriate containers and allow to pump through manifold until a stable baseline is achieved.

11.9 Select Run Tray.

11.10 Check the linearity and replication of the calibration curve. Acceptance criteria are $r \geq 0.995$. If calibration is acceptable, continue with sample analysis. If calibration is not acceptable, recalibrate.

## 12.0 PROCEDURE

12.1 Place samples on the autosampler tray and continue with analysis as described in the Calibration section.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 After review, enter final results into the LIMS system.

13.2 The instrument calculates the concentration of an aqueous sample by comparison against the calibration curve. Calculate the sample concentration of solid samples as follows:

$Cr^{+6}$, mg/kg = (A) (B) / C

where: A = concentration measured in mg/l
B = final volume of digestate, ml
C = sample size, g

## 14.0 METHOD PERFORMANCE

14.1 Method Detection Limit

The latest MDL study yielded the following data:

| | | |
|---|---|---|
| n = | 7 | |
| Standard Deviation ($\sigma_{n-1}$) | | ug/l |
| Spiked Concentration | | ug/l |
| Average Concentration | | ug/l |
| Average Recovery | | % |
| Calculated MDL | | ug/l |

14.2 Initial Demonstration of Capability

A typical IDC study will yield data similar to:

| | | |
|---|---|---|
| n = | 4 | |
| Standard Deviation ($\sigma_{n-1}$) | | ug/l |
| Spiked Concentration | | ug/l |
| Average Concentration | | ug/l |
| Average Recovery | | % |

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Dispose of any resulting residue, digestate, or extract in accordance with local sanitary regulations.

16.2 Additional sample shall be disposed of properly following the completion of analysis and an appropriate additional holding time.

## 17.0 REFERENCES

17.1 SW-846 Method 7196A

17.2 Standard Methods Method 3500-Cr D, 18th ED

17.3 SW-846 Method 3060A

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

July
~~May~~ 2002

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# QUALITY ASSURANCE PROJECT PLAN
# J-PIT REDEVELOPMENT SITE
# GARY, INDIANA

*Prepared for*

CITY OF GARY

**Department of Environmental Affairs**

***504 Broadway, Suite 1012***

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## SIGNATURE AND APPROVAL PAGE

## QUALITY ASSURANCE PROJECT PLAN and SAP
## FOR
## FIELD INVESTIGATION
## AT THE
## J-PIT REDEVELOPMENT SITE

**CITY OF GARY**
**GARY, INDIANA**

**REVISION 1**
**JULY 2002**

**Prepared for:**
**City of Gary**
**Gary, Indiana**

**Submitted by:**
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J-PIT REDEVELOPMENT SITE
PHASE II SITE INVESTIGATION
QUALITY ASSURANCE PROJECT PLAN

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## APPENDICES

**Appendices**

## F ACRONYMS

| | |
|---|---|
| %R | Percent Recovery |
| %RPD | Relative Percent Differe |
| BAKER | Baker Environmental, In |
| CLP | Contract Laboratory Pro |
| COC | Chain of Custody |
| D | Difference |
| DQO | Data Quality Objectives |
| EB | Equipment Blank |
| FB | Field Blank |
| FTL | Field Team Leader |
| GC/MS | Gas Chromatograph/Ma |
| HASP | Health and Safety Plan |
| IDEM | Indiana Department of E |
| IDW | Investigation Derived W |
| LCS | Laboratory Calibration S |
| LEL | Lower Explosive Limit |
| LIMS | Laboratory Information |
| LUSTS | Leaking Underground S |
| MS | Matrix Spike |
| MSD | Matrix Spike Duplicate |
| MW | Monitoring Well |
| NIST | National Institute of Sta |
| PAOCs | Potential Areas of Conc |
| PARCC | Precision, Accuracy, Re |
| Phase I ESA | Phase I Environmental S |
| Phase II ESA | Phase II Environmental |
| PID | Photo-Ionization Detect |
| PVC | Polyvinyl Chloride |
| QA | Quality Assurance |
| QAPP | Quality Assurance Proje |
| QC | Quality Control |
| RCRA | Resource Conservation |
| RD | Relative Difference |
| REC | Recognized Environmen |
| RW | Residential Well |
| SAP | Sampling and Analysis P |
| SB | Soil Boring |
| SCS | Soil Conservation Servic |
| SIMA | Simalabs International |
| SOP | Standard Operating Proc |

| | |
|---|---|
| SVOC | Semi-Volatile Organic C |
| SW | Surface Water |
| TB | Trip Blank |
| TCE | Trichloroethane |
| TRI | Toxic Release Inventory |
| USDA | United States Departme |
| USEPA | United States Environm |
| USGS | United States Geologica |
| VOA | Volatile Organic Analys |
| VOC | Volatile Organic Compo |
| VRP | Voluntary Remediation |

# SECTION 1.0

# 1.0 OVERVIEW

In May 2000, the City of Gary received a Brownfields Assessment Demonstration Pilot Grant from the United States Environmental Protection Agency (USEPA). The goal of the Pilot is to work with the City's partners to identify, assess, and redevelop local brownfields within the approximately 200-acre J-Pit Redevelopment (Sites Site), which are located within the 8,200 acre Airport Development Zone. The area is comprised of the approximately 100 acre Greenspace Site (a former sand mine known as the J-Pit), and about 100 adjacent acres of abandoned and undeveloped property (Pilot Site). The J-Pit site is at the center of a proposed light industrial, commercial and greenspace complex. The proposed restoration plan for the J-Pit is to fill it with groundwater to form a lake. The banks of the new lake would be restored through plantings and construction of wetlands, and a trail would be constructed around the lake, which would connect to the industrial/commercial complex and a new city park adjacent to the site.

This document presents the Quality Assurance Project Plan (QAPP) for the Limited Phase II Environmental Site Assessment (Phase II ESA) activities at the J-Pit Redevelopment sites (the Site) located in the City of Gary, Lake County, Indiana. The purpose of this field investigation is to perform a Limited Phase II Environmental Site Assessment (Phase II ESA). The QAPP has been prepared on behalf of City of Gary by Baker Environmental, Inc. (Baker). Integral to the QAPP are the Health and Safety Plan (HASP), the Sampling and Analysis Plan (SAP), the field standard operating procedures (SOPs), the laboratory accreditation and laboratory SOPs, and the Resumes of Key Personnel are included in Appendices A, B, C, D, and E respectively. The distribution list is provided in Table 1-1.

## 1.1 Project Organization

The elements of the quality assurance and management responsibilities of key project personnel including the organization structure are defined below. The organizational structure and lines of authority of the key personnel for the City of Gary J-Pit Redevelopment Project are outlined in Figure One.

## Project Management

### U.S. EPA - *Project Manager and QA Reviewer*

The U.S. EPA Brownfield Project Manager and QA Officer for this project is Jan Pels. Ms. Pels will be responsible for Agency Coordination and oversight for the project as well as oversight of quality assurance and laboratory issues.

### City of Gary

*Project Manager*

The City of Gary Project Manager will be Ms. Mary Mulligan. Ms. Mary Mulligan, the brownfield specialist for the City, is responsible for project direction concerning technical issues and strategies, setting work assignments for consultants, report writing and budgeting.

*Coordinator*

Dorreen Carey will assist Ms. Mulligan with project direction particularly with the greenspace development and wetland issues.

### Baker Environmental

*Baker Project Manager*

The Baker Project Manager will be Rick Spitaler. Mr. Spitaler has overall responsibility for ensuring that the project meets USEPA and IDEM objectives and Baker's quality standards. The Baker Project Manager will report directly to the City of Gary and is responsible for monitoring technical, cost and schedule performance. Mr. Spitaler will also be responsible for overseeing the writing of the report and provide oversight of health and safety.

*Baker Technical Manager*

The Baker Technical Manager for the project will be Jim Peyton. Mr. Peyton will assist the Project Manager with the project design and costing.

*Baker Quality Assurance Manager*

The Baker QA Manager is Mr. Thomas (Tom) Noble. Tom is responsible for ensuring that all regulatory and Baker procedures for this project are being followed. The QA Manager is also responsible for review of data, auditing the implementation of the QA program, reporting on adequacy and status of the program. It should be noted that the Baker QA Manager is outside the line of management. This allows him the ability to immediately address any noncompliant concerns that arise. The Baker QA Manager will perform Field Audits during on-site activities.

Data validation will be conducted by ECT Consultants. Following receipt of the validation report, the Baker QA Manager will perform a data assessment.

*Baker Data Management Officer*

Baker Environmental Data Management Officer, Ms. Margaret (Peggy) James, will act as the data management officer for the project and will be responsible for the organization and compilation of data including computerized formats.

*Baker Field Team Leader*

The Baker Field Team Leader is the Project Manager for this project. The Field Team Leader (FTL) is responsible for leading and coordinating the day to day activities of various resource specialists. The FTL will be responsible for implementing The sites Health and Safety Plan.

*Field Technical Staff*

The Technical Staff for this project will be drawn from Baker's pool of resources in the Merrillville, Indiana Office. The technical team staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work. The sampling team will assist the Field Team Leader as needed with sample collection, packaging, and calibration of field instruments.

*Additional Services*

Additional support services including hydrogeologists, engineers, and risk assessors are available on an as needed basis from Baker's Chicago Office and Corporate Headquarters in Pittsburgh.

**V-3 Consultants**

*V-3 Project Manager*

The V-3 Project Manager will be Mr. R. Damon Lee. Mr. Lee will act as co-manager in development and writing of Phase II ESA reports and will be responsible for wetlands assessment and site surveying. Mr. Lee will report directly to the City of Gary.

*V-3 Wetlands Assessment Team Leader*

The wetlands Assessment Team Leader will provide an assessment of wetlands issues and will be directly responsible to the V-3 Project Manager.

*V-3 Survey Team Leader*

V-3 Survey Team Leader and staff will be responsible for surveying of wells and other selected sample points for the Phase II ESA and the wetlands assessments and will be directly responsible to the V-3 Project Manager.

**Laboratory - Sima International Laboratory (Sima)**

*Laboratory Project Manager*

Sima Laboratory Project Manager, Ms. Michelle Dilley, will be responsible for ensuring that the laboratory fulfills the analytical needs of the project. She will ensure that the laboratory performs the QA/QC in accordance with Data Quality Objectives. The Project Manager will be responsible for ensuring resources of the laboratory are available on an as-required basis and provide an overview of final analytical reports

*Quality Assurance Manager*

Jeff Loewe is the Quality Assurance Manager for Sima Laboratories. Mr. Loewe will be responsible for making sure that the necessary systems are in place to meet the project data quality objectives, as defined by this QAPP. He will also be responsible for evaluating data quality, laboratory audits, performing data reviews, and documenting training. The QA officer has the overall responsibility for data after it leaves the laboratory. The QA officer will be independent of the laboratory management but will communicate data issues through the Laboratory Project Manager.

## 1.2 Facility History/Background Information

The following subsections provide a description of the site regarding location, history, and previous investigations.

### 1.2.1 Facility History

The area is comprised of the approximately 100 acre Greenspace Site (a former sand mine known as the J-Pit), and about 100 adjacent acres of abandoned and undeveloped property (Pilot Site). The approximately 100 acres Pilot Site property is comprised of four individual sections that are located east of Burr Street south of 15th Avenue, west of the E.J.& E. Railroad and north of 23rd Avenue in Gary (Calumet Township), Lake County, Indiana. The site is within Sections 11, 12, 13 and 14, Township 36 North, Range 9 West of the Third Principal Meridian. The site is in an area of mixed usage including residential subdivisions, undeveloped properties, and some industrial areas (Figure 3-1).

In July 2001, Phase I ESAs were performed by Environmental Design International, Inc. for the approximate 100 acre J-Pit Greenspace Site and adjacent Pilot Site parcels. The Pilot Site is comprised of four Sections as described below:

- Section 1: This section consists of approximately 20 acres and is bound on the west by Hobart Street, on the north by 15th Avenue, on the east by Dallas Street, on the southeast by the closed Gary Municipal Landfill and on the southwest by the J-Pit.

- Section 2: This section consists of approximately 15 acres south of the J-Pit and is bound on the south by 21st Avenue, on the west by Colfax Street, on the north by 22nd Avenue, and on the east by Fairbanks Street. The eastern fifth of the section contains a parcel formerly occupied by an auto scrap yard.

- Section 3: This section lies directly south of the closed Gary Landfill and consists of approximately 30 acres bound to the east by Colfax Street, Hamlin Street and King Street; to the south by 21st Avenue; to the north by 23rd Avenue; and to the west by Calhoun Street.

- Section 4: This section encompasses roughly 40 acres and is bound to the north by 21st Avenue, to the south by 23rd Avenue, to the west by Fairbanks Street, and to the east by the EJ&E Railroad line.

Individual Phase I ESA reports were prepared for each of the Pilot Site Sections and the Greenspace Site (i.e. five total reports). Several Recognized Environmental Concerns (RECs) were documented in the Phase I ESA Reports. In general, some level of Phase II sampling was recommended for all five parcels due to their proximity to the closed Gary Municipal Landfill, two Superfund sites (Midco I and 9th Avenue Dump), several Leaking Underground Storage Tank (LUST) sites, and specific areas judged to constitute on-site RECs as outlined below.

- Section 1: Although the parcel is predominantly vacant, two small buildings were observed on the west side (south of 15th Avenue), an area historically used as a junk yard. Surface and buried refuse, tires, an old bus, metal, plastic, and empty 55-gallon drums were observed.

- Section 2: Structures and scattered piles of debris including shredded tires, two empty 55-gallon drums, and scrap metal were observed on the northeast side in the area of the former Paul's Auto Yard (a scrap yard once located at 2124 Colfax); and LeRoy's Used Cars (previously located at 2150 Colfax). In addition, scattered rubbish was observed along the boundary line with the J-Pit area to the north.

- Section 3: The north and northwest portions of this parcel were reportedly used in the past for car repairs and dumping. Prior to 1990, the Gary Municipal Landfill did not have a slurry wall along the approximately 1,200 foot common property line. Active junkyards are located adjacent to the parcel southwest. Bivona, Inc., to the southeast, was listed on the Toxic Release Inventory (TRI) for Trichloroethane (TCE) and currently is enrolled in the IDEM VRP. Debris, scattered piles of rubbish including shredded tires, and stressed vegetation were observed at various locations throughout the site.

- Section 4: Partially buried construction debris was observed near the boundary fence of the J-Pit (north border); and railroad tracks are located along the west border.

- Greenspace Site (J-Pit): At one time this site was used as a waste area operated by Waste Management. An oily, rust-colored unknown substance was observed in an approximately 1,300 foot excavated ditch, located on the north side. Debris was observed along the perimeter including tires, concrete piping, concrete, scrap metal, plastics, and some buried debris. Three partially submerged monitoring wells were observed in water on the east side, as well as one well in the southeast corner and one in the northeast corner of the site. A groundwater and leachate collection system for the closed Gary Municipal Landfill is located on the east side. The system piping is seven feet in diameter, with three inlets from the south, north and northeast. Inlet piping runs under vegetation in a ditch extending along the east end of the parcel. The west wall of this ditch is used as a service road. Stressed vegetation was observed along the wall of the ditch, five feet from the bottom.

As part of the evaluation of site background, Baker reviewed aerial photographs from 1938, 1958, 1965, 1973, 1987 and 1992, acquired from the records of the Soil Conservation District for Lake County, Indiana. In 1938, the Site was part of a largely undisturbed dune and swale topography typical of the majority of the region. By 1958, the J-Pit sand mine (Section 5) and the Gary Municipal Landfill were visible and apparently active. Subsequent photographs indicate periodic flooding of the J-Pit and the emerging industrial and semi-industrial development of the surrounding vicinity. The surrounding

properties (Sections 1 to 4) remained largely undeveloped except for the construction of pre-subdivision development roadways in Section 2 and some businesses along Colfax Avenue.

### 1.2.2 Site Geology/Hydrogeology

According to the 1992 United States Department of Agriculture (USDA) Soil Conservation Service (SCS) Soil Survey of Lake County, Indiana, the site and immediate area are underlain by the Oakville-Tawas complex and the Tawas Muck soils. The Oakville-Tawas complex is comprised of about 45 percent Oakville sand and 45 percent Tawas Muck. The Oakville sand is a black to yellowish brown, excessively drained, fine sand located on narrow ridges. The Tawas muck is a black, deep, very poorly drained organic muck underlain with a grayish-brown loose sand in depressions between the Oakville sand ridges (1992, USDA, Lake County Soil Survey). The surface at the site has been heavily altered by sand mining, filling, grading, spreading gravel and stockpiling activities.

The site is located at the boundary of lacustrine deposits of the Calumet Lacustrine Plain physiographic region and the Toleston Beach complex. The Calumet Lacustrine Plain has been heavily altered due to industrial and residential development. Where undisturbed, the area is dominated by three relict dune-capped beach ridges separated by broad inter-ridge marshes. The Toleston Beach complex extends from between the Little Calumet River and the Grand Calumet River to Lake Michigan and is characterized by linear ridges of unified parabolic dunes interspaced with interdunal swamps (dune and swale). The unconsolidated materials were deposited in the Wisconsin age, with a combined action of ice, wind and water. The glacial deposits in the vicinity and surrounding area have an estimated thickness of 150 feet (1994, Department of Environmental Resources Water Resource Availability in the Lake Michigan Region, Indiana).

The site is within the Calumet Aquifer System consisting of fine to medium sand with beach gravel beds. Discontinuous beds of silt, clay and peat deposits confine the aquifer in some locales. The aquifer is considered highly susceptible to surface contamination due to the lack of a clay cap or separator beds (1994, Department of Environmental Resources Water Resource Availability in the Lake Michigan Region, Indiana). Regional groundwater flow is anticipated to be to the north toward

the Grand Calumet River and Lake Michigan, but due to the location of the site between the Grand Calumet and Little Calumet Rivers, local groundwater flow may differ.

The unconsolidated soils are underlain by the Wabash Formation within the Kankakee Arch. The Wabash Formation is comprised of reef and inter-reef Silurian dolomite, dolomitic limestone and argillaceous limestone (1994, USGS Hydrogeologic Atlas of Aquifers in Indiana).

## 1.3 Project Objectives

The City of Gary's goal is to assess and develop the Site as part of the overall Airport Redevelopment Zone. The goals of the Phase II ESA is to provide sufficient information with respect to the nature and extent of contamination to assist in the redevelopment of the site. As part of the Phase II ESA the following will be evaluated:

- Identify affected media (soil, groundwater, surface water and sediment);
- Identify Contaminants of Concern;
- Delineate the vertical and horizontal extent of contamination in each medium;
- Determine potential human and ecological receptors and exposure pathways; and
- Provide sufficient information to make preliminary decisions on remedies and default and non-default closure options for the source area(s).

In addition the Phase II ESA is to provide a determination for what additional areas may require further investigation or may benefit from remedial action or risk assessment.

To achieve these project objectives, soil, groundwater, sediment and surface water samples will be collected from these areas. Field and laboratory protocol will ensure that the data are technically and legally defensible. All analytical data will be of quality such that it may be used quantitatively.

## 1.4 Data Quality Objectives

The Data Quality Objective (DQO) Process is series of planning steps based on the Scientific Method that is designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for intended application. The steps of DQO process are included in Figure 3 of this QAPP.

DQO are qualitative and quantitative statements derived from outputs of each step of the DQO Process that:

- Clarify the study objective;
- Define the most appropriate type of data to collect; and
- Determine the most appropriate conditions from which to collect the data.

The DQO are then used to develop a scientific and resource-effective sampling design.

The DQO Process allows decision makers to define their data requirements and acceptable levels of decision during planning before any data are collected. DQO were based on the seven-step process described in EPA QA/G-4 (September 1994) document and outlined in Figure 3.

The data quality objectives for this project are summarized in Table 1-2 and detailed below.

### 1.4.1 Problem Definition

The specific objectives of the Phase II ESA are to evaluate the recognized environmental conditions identified in the Phase I ESA and those determined during Pre-field Phase II activities. As indicated in the Phase I ESA, several areas within each Section of the site were judged to constitute recognized environmental concerns. In addition, the site is bordered by, or in the vicinity of, several properties with recognized environmental concerns. The potential areas of concern (PAOCs) for each Section include the following:

Section 1

- Scattered illegal dumping
- Potential impact from down-gradient trucking facilities

Section 2

- Impact from former scrap yard operations
- Scattered illegal dumping

Section 3

- Impact from off-site landfill and Bivona VRP site
- Scattered illegal dumping

Section 4

- Scattered illegal dumping
- Potential impact from side gradient trucking facilities

Section 5 (J-Pit)

- Impact from off-site landfill
- Scattered illegal dumping
- Prior operations at J-Pit
- Potential impact from side and down gradient trucking facilities

### 1.4.2 Identify the Decision

To proceed with the development process, the following will need to be determined:

- Are contaminants present above human and ecological risk-based exposure levels in soil, sediment, surface water and groundwater?
- Has a release occurred at areas identified in Phase I ESA and during ongoing prefield activities?
- Is there migration of impacted groundwater from the landfill?
- Is there an immediate or acute hazard?

### 1.4.3 Identify Inputs to the Decision

The project goals are to meet the VRP residential and non-residential clean-up criteria, as defined in the Voluntary Remediation Guidance Document, July 1996. The specific VRP cleanup criteria are the identified contaminants of concern and others as defined in Table1-2. In order to proceed with development, several inputs to the decision process will need to be addressed. These include:

1. Develop a sampling plan that will obtain adequate samples to be representative of the concentration of residual constituents at the Site.
2. Ensure that analytical methods are adequate to detect levels of constituents in media of concern at or below the IDEM VRP-based screening criteria.

### 1.4.4 Define Boundaries

The spatial boundaries of the Site consist of five Sections totaling over 200 acres (described in preceding Sections). The J-Pit area is bound to the east by the Gary Municipal Landfill and Calhoun Avenue, to the west by the EG & E railroad tracks, to the north by Fifteenth Avenue and to the south by 23rd (see Figure 2). Internally, boundaries for PAOC are broken down into the following categories:

- *Areas known to be contaminated*
  Areas identified in Phase I ESAs, scrap yard in Sections 1 and 2, J-Pit bermed area, groundwater from Bivona and the Landfill,
- *Areas that may be contaminated*
  J-Pit surface water impoundment, scattered dumping on north end of Section 3, along undeveloped streets in Section 2, north end of Section 4.
- *Areas unlikely to be contaminated*
  Section 1, Section 4, J-Pit west of berm, west portion of Section 3, west portion of Section 4.

Seasonal data are not required to determine the presence or absence (i.e., release) of a constituent in the soil or sediment. Groundwater data would be collected quarterly to address temporal boundaries for evaluation of potential groundwater evaluations.

Practical constraints that may impede sampling will be considered (e.g., foundations, utility lines or process equipment).

The potential pathways for PAOC may include the following:

- Unlined J-Pit: air emissions, direct contact, contamination of soils, and infiltration to groundwater.
- Scattered illegal dumping: Air emissions, direct contact, contamination of soils, and infiltration to groundwater.
- Operations at the auto and scrap yards in Section 1: air emissions, direct contact, contamination of soils, and infiltration to groundwater.
- Off-site groundwater impacts: air emissions, direct contact, contamination of soils, and infiltration to groundwater.

### 1.4.5 Develop a Decision Rule

Based on the results of the Phase II ESA, the following objectives will be reviewed to determine a decision rule.

| Objective | Decision Rule |
|---|---|
| Were chemicals released to the environment from the potential sources? | Yes: The PAOC continues in the VRP process<br>No: No further action is required. |
| Are the released chemicals present in the environmental media above VRP-based screening criteria? | Yes: The PAOC continues in the VRP process<br>No: No further action is required. |
| Do the quantities of chemicals present indicate a potential risk to human health or the environment? | Yes: The PAOC continues in the VRP process and the extent of contamination will be delineated.<br>No: No further action is required. |

If the entire site does not meet VRP criteria, then potential sections of the property may meet VRP requirements and other portions may require further review and definition of boundaries.

If the entire site does not meet VRP criteria, then the Phase II ESA will be reviewed to further define areas of concern and define extent, fate and transport of chemical of concern within each area.

### 1.4.6 Specify Limits on Decision Errors

The possibility of decision error may occur as sampling design error and/or measurement error. Although the possibility of decision error can never be totally eliminated, it can be minimized and controlled. For this project, sampling and measurement decision errors are to be controlled. The VRP default sampling scheme includes representation of the potential source release areas at the PAOC.

Measurement errors are controlled through the use of analytical methods that achieve reporting limits less than the conservative default remediation standards. The following describes the possible decision errors and potential consequence of each error:

- Decision error based on a false positive (i.e., the chemical concentration is identified as greater than the VRP clean up objectives when in fact it is less than the standard). If this occurs, additional characterization of the PAOC would be conducted when it is not warranted. This will incur additional costs and potentially delay the schedule. This error, however, will ensure compliance with the site-specific remediation standards and protection of human health and the environment.

- Decision error based on a false negative (i.e., The chemical concentration is identified as less than the site-specific remediation standard when in fact it is greater than the standard). If this occurs, additional characterization that is warranted would not be performed. This error may lead to soil with chemical concentrations greater than the site-specific remediation standards left in place. The likelihood of a decision error based on a false negative is minimal for the following reasons:

  The sampling program is biased toward known or suspected PAOC areas where there is the highest probability of encountering a potential release.

Samples will be collected per the VRP clean-up objectives at multiple locations in each PAOC to ensure adequate positive-bias sampling of the potential source area. The analytical program will be designed to be sensitive enough to record chemical concentrations below the VRP screening levels as defined by IDEM VRP.

### 1.4.7 Optimize the Design for Obtaining the Data

In order to ensure obtaining usable quality data with the limited available historical data, reports, etc., the City of Gary has elected to use IDEM VRP sampling approaches with selection of the most appropriate VRP clean-up goals upon known or suspected site conditions.

Effective measures include the use of Geoprobe® sample acquisition for the majority of the field sampling, use of test pits to target the PAOC and sample locations while still meeting the DQO objectives. Additional field screening methodologies (VOCs screened with a photoionization detector) can be very cost effective, but only if the potential parameter suite can be realistically reduced based on site data.

Sampling rationale and locations are located in Appendix B (SAP). Sample identification, number, parameters and frequency are in Table 1-3 in Appendix B.

## 1.5 Measurement Performance Criteria

The PARCC parameters (precision, accuracy, representativeness, comparability, and completeness) are qualitative and/or quantitative statements regarding the quality characteristics of the data used to support project objectives and ultimately environmental decisions. These parameters are presented in this section.

### 1.5.1 Precision

Definition: Precision is a measure of the degree to which two or more measurements are in agreement.

#### 1.5.1.1 Field Precision Objectives

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 20 or fewer analytical samples per matrix. The frequency of matrix spike/matrix spike duplicates (MS/MSDs) that are collected in the field for laboratory precision purposes will be collected at a rate of one MS/MSDs per 20 analytical samples per matrix. The total number of duplicates and MS/MSDs for this project are found in Summary Table of Sampling and Analysis Program (Table 1-4).

#### 1.5.1.2 Laboratory Precision Objectives

The degree of agreement between the numerical values of a set of duplicate samples performed in an identical fashion constitutes the precision of the measurement. Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD samples for organic analysis .and laboratory duplicate analyses for inorganic analysis. Precision will be reported as a relative percent

$$\%RPD = \frac{S - D}{(S + D)/2} X\ 100$$

difference and will be calculated for each pair of duplicate analysis using Equation 13-2.
Where:

% RPD = Relative percent difference.

S = First sample value (MS for organics and initial sample result for inorganics).

D = Second sample value (MSD for organics and method duplicate for inorganics).

1.5.2 Accuracy

1.5.2.1 Definition:

Accuracy is the degree of agreement between an observed value and an accepted reference value.

1.5.2.2 Field Accuracy Objectives

Accuracy in the field will be assessed through the use of equipment rinsate samples and trip blanks and through the adherence to all sample handling, preservation and holding times.

1.5.2.2 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of matrix spikes of standard reference materials and the determination of percent recoveries. Analytical accuracy is expressed as the percent recovery of an analyte that has been added to the sample or standard matrix (i.e., blank) at a known concentration before analysis. The percent recovery of matrix spike samples will be calculated using the following equation:

$$\%R = \frac{A - B}{C} X\ 100$$

% R = Percent recovery

A = The total analyte concentration determined experimentally from the spiked sample.

B = The background level determined by separate analysis of the unspiked sample.

C = Amount of the spike added.

Accuracy control limits are given in Table 1-2 and included in the SOPs in Appendix D. During the course of the Phase II ESA, these objectives may be revised with client approval in light of matrix interferences or other factors not fully accounted for in the EPA Methods and laboratory SOPs.

### 1.5.3 Completeness

#### 1.5.3.1 Definition:

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was planned to be obtained under normal conditions.

#### 1.5.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the field during the RD for each parameter in a given matrix. The equation for completeness is presented below.

$$Completeness = \frac{Valid\ Data\ Obtained}{Total\ Data\ Planned} X\ 100$$

The field completeness goal for this RD is 90 percent or greater.

#### 1.5.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the laboratory during the project for each parameter in a given matrix. The equation for completeness is presented above. The laboratory completeness goal for this project is 95 percent or greater.

### 1.5.4 Representativeness

#### 1.5.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

#### 1.5.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program. To ensure a representative soil sample after the collection of VOC samples all soil will be placed in a stainless steel bowl and homogenized prior to filling the respective laboratory-prepared jars.

After the purging of each well volume pH, specific conductivity, and temperature measurements will be recorded. Once three consecutive measurements are within ten percent of each other groundwater samples will be collected. These procedures are conducted in order to ensure representativeness of the samples.

For complete discussion of sampling refer to the SAP (Appendix B) and associated field sampling and field instrument Standard Operating Procedures (SOPs Appendix C).

#### 1.5.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory will be satisfied by using the proper analytical procedures, meeting sample holding times and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of site conditions. During development of this network, consideration was given to past waste management practices, existing analytical data, and physical setting and processes. The rationale of the sampling network is provided in Section 2.4 of the QAPP and in the SAP (Appendix B).

1.5.5 Comparability

1.5.5.1 Definition:

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is also dependent on similar QA objectives.

1.5.5.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the SAP is followed and that proper sampling techniques are used.

1.5.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented in the QAPP. Comparability is also dependent on similar QA objectives.

1.5.6 Level of Quality Control Effort

Equipment rinsates, trip blanks, method blanks, duplicates and standard reference materials including matrix spike and surrogate samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Equipment rinsates and trip blanks consist of analyte-free water which will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Equipment rinsate samples are analyzed to check for equipment decontamination procedures which may cause sample contamination. Trip blanks will be used in conjunction with off-site aqueous VOC samples to assess the potential for cross-contamination during sample shipment and storage. Trip blanks prepared prior to the sampling event in the actual sample containers and are kept with the investigative samples throughout the sampling event. They are then packed for shipment with other samples and sent for analysis. There should be one trip blank included in each sample shipping

container that has aqueous VOC samples. At no time after their preparation are the sample containers for trip blanks opened before they reach the laboratory.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One MS/MSD will be collected for every 20 samples per matrix. MS/MSD samples are designated/collected for metals and organic analyses.

MS/MSD samples are investigative samples: that is, actual matrix sample is collected in the field. Soil MS/MSD samples require no extra volume for VOCs or inorganics. However, aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for inorganics. One MS/MSD sample will be collected or designated for every 20 investigative samples per sample matrix (i.e., groundwater, soil).

The general level of the QC effort will be one field duplicate per 10 investigative samples per sample matrix and one equipment rinsate blank per 20 samples per sample matrix. One VOC trip blank consisting of analyte-free reagent grade (Type II) deionized water will be included along with each cooler of aqueous VOC samples.

The number of QC samples to be collected for Section 2.5. Sample collection procedures for the QC samples are specified in Section 3.0 of the SAP (Appendix B) and the SOPs in Appendix C. Table 1-2 contains the reporting limits for organic and inorganic parameters for the off-site laboratory.

## 1.6 Special Training Certification and Records

All personnel will be trained in accordance with OSHA's 29CFR 1910.120 regulation covering Hazardous Waste Operations and Emergency Response. Further discussion of health and safety training is provided in Appendix A. No additional training or certification is required for the sampling conducted during the Phase II Investigation.

## 1.7 Documentation and Records

The QAPP project data and information will be documented, tracked and managed from their generation in the field to final use and storage in a manner that ensures data integrity and defensibility. All field data will be documented in field log books and all laboratory records maintained. Further description of field, laboratory and final evidence file documentation is described in Section 2.4 (Sample Handling and Custody Requirements) and outlined in the F300 Documentation Section of the SOPs in Appendix C of the QAPP. Included in this Section are the following: Sample Preservation and Handling (F301), Chain of Custody (F302), Field Logbook SOP (F303) and QA/QC Samples (F304).

## 1.8 Schedule

See Figure 4 for the Project Schedule.

SECTION 2.0

# 2.0 DATA GENERATION, ACQUISITION AND REPORTING

## 2.1 Sampling Process Design

The sampling procedures to be used in for this project are consistent with the objectives of this project. Please refer to Section 3.0 of the SAP located in Appendix B. The SAP outlines all the sampling design and procedural information.

## 2.2 Analytical Methods Requirements

The project laboratory will implement the project required analytical SOPs. These laboratory SOPs for sample preparation and analysis are based on SW-846 Third Edition, June 1997, and for all final updates. Laboratory Specific SOPs have been developed for each routine analytical procedure. These SOPs detail the steps performed at SIMA LABs. The analytical specific SOPs for analysis are included as Appendix D. These include Aqueous and Non-Aqueous sample preparation and sample analysis for Organic and Inorganic analysis.

## 2.3 Sampling Methods Requirements

### Sampling

During the Phase II ESA surface soil, subsurface soil, groundwater, surface and sediment samples will be collected. Collection methods and procedures that will be used during the investigation are defined in the following groups of SOPs. Field SOPs are provided in Appendix C of the QAPP.

Drilling and Sampling methods are defined in SOPs: F101 (borehole and sample logging), F102 Soil and Rock Sample Acquisition, F103 Monitor Well Installation, F104 Groundwater Sample Acquisition, F105 Surface Water and Sediment Acquisition, F106 Test Pit and Trench Excavation, , F107 Wastewater Sample Acquisition, F108 Drum Sampling.

Decontamination and Waste Handling F501Decontamination of Drilling Rigs and Monitoring Well Materials, F502 Decontamination of Sampling Well and Monitoring Equipment, F504 Handling of Site Investigation Wastes

Field Testing, maintenance and inspection requirements F401 Aquifer testing, F402 Slug Testing, F201 On-site Water Quality Testing, F201Water level, F202 Water –Product Level, and Well Depth Measurements, , F203 Photoionization detector, F204 Flame Ionization detector.

Pre-preserved or non-preserved sample containers will be supplied by the laboratory (Table 2-1) and will be level 1 certified pre-cleaned sample containers, which are QC checked by the manufacturer.

## 2.4 Sample Handling and Custody Requirements

Custody is one of several factors which is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, will be maintained under document control in a secure areas at Baker Environmental, Inc. Merrillville, Indiana and at the City of Gary.
A sample or evidence file is under your custody if:

- The item is in actual possession of a person
- The item is in the view of the person after being in actual possession of the person
- The item was in actual physical possession but is locked up to prevent tampering
- The item is in a designated and identified secure area

For this project, proper chain-of-custody documentation will be maintained for all samples from the time of collection until they are shipped to the laboratory. Chain-of-custody sheets accompanying the samples will contain the following information: project number, sampler(s) name, sample members, number of containers, method(s) of preservation of samples, date and time of sample collection, analysis(es) requested, date and time of transportation to the laboratory, method of transportation, and

any other information pertinent to the samples. Sample documentation will be prepared in Baker's SOP for sample chain-of-custody is included in Appendix C (F302).

**Field Custody Procedures**

Field logbooks will provide the means of recording data collection activities. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned
- Logbook number
- Project name
- Project start date
- End date

Entries into the logbook will be made in indelible ink. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in indelible ink, signed, and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark which is signed and dated by the sampler. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station shall be

recorded. The number of the photographs taken of the station, if any, will be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the SAP in Appendix B and the SOPs in Appendix C and Table 1-3. The equipment used to collect samples will be noted, along with the time of sampling, sample description, and depth at which the sample was collected. Sample site-specific identification number will be assigned prior to sample collection. The site-specific sample number should consist of the following:

- Subsurface soil samples will be designated with a SB prefix (for soil boring) and boring number followed by the individual sample number from that location. An example of this would be SB01-01, where SB indicates a soil boring, 01 indicates that first boring in the series, and –01 designates the first sample collected from that boring. Residential water samples will be indicated by RW prefix.

- Monitoring wells installed during the investigation will have an MW prefix followed by the number designation. Groundwater samples collected from the initial sampling event, conducted after the installation of the new wells, will be simply designated by the well number (i.e., MW-01).

- The surface water sample to be collected will have an SW prefix and sediment will have a SD prefix.

- Sample from which QA/QC samples were collected will have the suffix of D for duplicate or MS/MSD for matrix spike/matrix spike duplicate attached to the sample designation. Equipment rinsates will be designated by the prefix RB and a sequential number while field blanks will be designated by a FB prefix and trip blanks with a TB prefix.

- The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain of custody intact. Examples of field custody procedures are provided below.

- The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.

- All bottles will be identified by use of sample labels with sample numbers, sampling locations, date/time of collection, and type of analysis.

- Sample labels are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample label because the ballpoint pen would not function in freezing weather.

- Samples are accompanied by a properly completed chain of custody form. The sample numbers and locations will be listed on the chain of custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

- Samples will be properly packaged on ice at 4°C for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in and secured to the inside top of each sample box or cooler. Shipping containers will be locked and secured with strapping tape and custody seals for shipment to the off-site laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations. Since shipping containers for the on-site lab will be hand delivered by the Field Team Leader, custody seals are not required.

- All shipments will be accompanied by the Chain of Custody Record identifying the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for returning to the sampling office.

- If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be

registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

- Samples will be present at the off-site laboratory within one day of sample collection. Samples collected and kept overnight (i.e., not sent to the lab the same day the sample was collected) will remain in the custody of the Field Team Leader. Samples will be refrigerated and secured.

Laboratory Custody Procedures

At Sima, the transfer of sample custody is documented on the COC. A completed COC must accompany all samples collected in the field for laboratory analysis. The sample receipt department receives all samples into the laboratory. All sample containers are visually inspected for bottle breakage and other abnormalities. Any abnormalities are documented in the LIMS "ChkList" feature. Any discrepancies between the sample container identification and the COC are noted in the LIMS. Client name, address, contact person, name and signature of sample collector, sample description, date and time of sample collection, number of containers, matrix, requested analysis and a relinquishing signature are required information on the COC. The LIMS "ChkList" form is used to document the carrier name, condition of the cooler, custody seals (if present), sample containers, presence of required COC information, chemical preservation, receipt temperature of samples, as well as if the samples are received chilled, on ice, in acceptable containers and within proper holding time. Information on rush samples or samples in danger of exceeding hold times are immediately forwarded to the appropriate Project Manager. The sample receipt temperature is measured using a temperature blank sample. If a temperature blank is not available, the measurement of a single representative sample container is performed. The acceptance criteria for receipt temperature are 0.1 - 6 C. Samples that are delivered within 2 hours of collection are considered acceptable if there is evidence that the chilling process has begun (e.g., ice or cooler packs are present). The sample control personnel sign the COC form prior to sample information being logged into the LIMS.

The Laboratory Project Manager will notify the client of any problem with a sample. This notification will be documented by the laboratory.

All samples received at the laboratory are logged into the LIMS. The LIMS assigns a unique laboratory sample number to each sample. Moreover, each container for a given sample is issued a unique container identification number.

Login personnel determine which analysis is required for a given sample from the information provided on the COC.

The sample COC, LIMS "ChkList" form and any other shipping paperwork are placed into a project file which is given to the Project Manager.

**Final Evidence Files**

The final evidence file will be the central repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. Baker is the custodian of the evidence file for the City of Gary and maintains the contents of evidence files for the site in a secured, limited access area and under custody of the Baker Project Manager. The information from the files will be available to the City of Gary upon request. After five years Baker will be responsible for archiving the files.

The final evidence file will include at a minimum:

- field logbooks
- field data and data deliverables
- photographs
- drawings
- soil boring logs
- laboratory data deliverables
- data validation reports
- data assessment reports
- subcontractor reports
- progress reports, QA reports, interim project reports

## 2.5 Quality Control Requirements

Quality control (QC) is the system of technical activities that measures the performance of a process. Different QC checks and samples are used to both prevent and identify specific sources of error in a particular process/activity. There are two general types of QC checks and samples:

- Batch-specific QC: These include QC samples that are handled, prepared and analyzed concurrently with environmental samples to ensure that the procedures used to collect, transport and analyze a group/batch of samples are performed under known, well-defined conditions.

- Sample-specific QC: These include QC samples that are used to evaluate potential sources of error in the collection, transport and analysis of individual samples.

The remainder of this section presents the QC checks and samples used for the post-excavation sampling program.

### 2.5.1 Field QC Requirements

Field measurements will be collected following the procedures outlined in Section 5 of SAP (Appendix B), Section 4.0 of this QAPP and SOPs in Appendix C. QC procedures for pH, Eh, conductivity, and temperature, are limited to checking the reproducibility of the measurement by obtaining multiple readings on a single sample or standard and by calibrating the instrument properly following manufacturer's instructions (Table 2-2). QC procedures for the HNu are limited to on site daily calibration according to the manufacturer's instructions. Calibrations standards will be recorded in the field logbook along with any corrective actions taken. All standards used for calibration must be from the NIST, traceable to NIST standards, or other accepted standards (e.g., USEPA).

Field duplicate, trip blank, equipment blank, field blank and cooler temperature blank will be analyzed to assess the quality of the data resulting from the field sampling program. Information gained from these analyses further characterizes the level of data quality obtained to support project goals. Each of

these types of field QC samples undergo the same preservation, analysis, and reporting procedures as the related environmental samples. Section 3.3 of the SAP (Appendix B) presents details on the procedures to obtain these QC samples. Each type of field QC sample is detailed below.

**Field duplicates** are two samples collected as a single sample, split and divided into two portions. Field duplicates are collected and analyzed for chemical constituents to measure the precision of the sampling methods used. The general level of the QC effort will be one field duplicate for every 20 or fewer investigative samples per medium.

**Trip blanks** will be submitted for analysis to provide the means to assess the quality of the data resulting from the field program. Trip blanks pertain to VOCs only. Trip blanks are used to assess the potential for contamination of VOCs resulting from contaminant migration into sample bottles/jars during sample shipment and storage. Trip blanks are prepared by the laboratory using organic-free reagent water before the sampling event. They are shipped to the site with the sample containers and kept with the investigative samples throughout the sampling event. They are then packaged for shipment with other VOC environmental samples and sent to the laboratory for analysis. At no time after trip blank preparation are the trip blank sample containers opened before they reach the laboratory. One trip blank will be included in each sample shipping container that contains VOC samples.

**Equipment blanks** are used to assess the effectiveness of decontamination procedures. Equipment blanks are obtained under representative field conditions by collecting the rinse water generated by running analyte-free water through sample collection equipment after sampling and decontamination and then placing the rinse water in the appropriate sample container for analyses. One equipment blank will be collected per each type of sampling equipment used at an area (e.g., trowel, clam bucket, etc.). It is anticipated that the backhoe will not be removed from the area until work is completed. If, however, the backhoe is removed from an area and then has to be remobilized to the area, another equipment blank will be collected. Equipment blanks are analyzed for the same chemical constituents as the associated environmental samples.

**Field Blanks** are used to assess the potential for background contamination from the ambient air. A sample will be obtained by filing a sample bottle with lab pure water, allowing it to sit uncapped while samples are being taken and then recapping and submitting the sample for analysis.

**Cooler temperature blanks** monitor the temperature of the cooler in which field samples are contained. The preservation requirement for many of the samples is to keep the sample temperature at 4°C. The cooler temperature blank allows for the measurement of this requirement. One cooler temperature blank will be included in each cooler with field samples.

The type of field QC samples and the required frequency of each type of sample is provided below.

| Field QC | Data Quality Indicator | Frequency |
|---|---|---|
| Field Duplicate | Precision | 1 field duplicate per 20 field samples per matrix |
| VOC Trip Blank | Contamination (Accuracy/Bias) | 1 trip blank per cooler that contains aqueous VOC samples. |
| Equipment Rinsate Blank | Contamination (Accuracy/Bias) | 1 equipment blank per sampling event per area per matrix |
| Field Blank | Contamination (Accuracy/Bias) | Frequency is based on the Professional Judgement of the Field Team Leader. |
| Cooler Temperature Blank | Preservation (Accuracy/Bias) | 1 cooler temperature blank per cooler |

These field QC samples will be handled in the same manner as the field samples.

## Laboratory Quality Control Checks

The laboratory (Sima) has a QC program used to ensure the reliability and validity of the analysis performed at the laboratory (see Appendix D). All analytical procedures are documented in writing as SOPs and each SOP includes a QC Section which addresses the minimum QC requirements for the

procedure. The internal quality control checks might differ slightly for each individual procedure but in general the QC requirements include the following:

- Field/Trip/Equipment blanks
- Method blanks
- Reagent/preparation blanks (applicable to inorganic analysis)
- Instrument blanks
- Matrix spikes/matrix spike duplicates
- Surrogate spikes
- Analytical spikes (Graphite furnace)
- Field duplicates
- Laboratory duplicates
- Laboratory control standards
- Internal standard areas for GC/MS analysis; control limits
- Mass tuning for GC/MS analysis

For a description of the specific QC requirements of this site investigation and the frequency of audit, refer to the laboratory SOPs (Appendix D). The QC criteria are also included in the SOPs.

All data obtained will be properly recorded. The data package will include a full deliverable package capable of allowing the recipient to reconstruct QC information and compare it to QC criteria. Any samples analyzed in nonconformance with the QC criteria will be re-analyzed by the laboratory, if sufficient volume is available. It is expected that sufficient volumes/weights of samples will be collected to allow for re-analysis when necessary.

## 2.6 Instrument/Equipment Testing and Maintenance Requirements

Proper maintenance of instruments and equipment is essential to ensuring their readiness when needed. Dependent on manufacturer's recommendations, maintenance intervals are established for each instrument/equipment. General maintenance activities are provided below.

**Field Equipment/Instruments**

The field equipment for this project include thermometers, pH meter, conductivity meter and PID. Specific preventative maintenance procedures for this equipment are discussed in the SOPs in Appendix C and Table 2-3, and will be conducted in accordance with the manufacturer's specifications.

Field instruments will be checked and calibrated daily before use. Calibration checks will be documented in the field logbook. Routine preventative maintenance as well as per use inspections and checkout will be conducted to assure proper operation of the various pieces of equipment (Table 2-4). The Field Team Leader will be responsible for implementing and documenting these procedures in the logbook.

**Laboratory Instruments**

At Sima, routine maintenance for all instrumentation is performed according to the manufactures recommended procedures (Table 2.5). The frequency of this maintenance is based upon the manufacturer's guidance and the experience of the trained analytical staff. Only trained staff and certified third party contractors may perform equipment and instrument maintenance and repairs. The use of manufacturer-recommended grades or better of supporting supplies and reagents is also a form of preventive maintenance. For example, gases used in the various gas chromatographs and metals instruments are of sufficient grade to minimize fouling of the instrument. The routine use of septa, chromatographic columns, and other supporting supplies from reputable manufacturers will assist in averting unnecessary periods of instrument downtime. An inventory of critical spare parts will also be maintained by the laboratory to minimize instrument downtime. Records of maintenance activities are maintained at the laboratory.

2.6.1 Instrument Calibration and Frequency

**Field Instruments**

Field instruments will be calibrated daily and as needed according to SOP F201and F203 for On Site Water Quality Testing and Photoionization Detector – PID, and according to manufacture's specifications. Calibration procedures, corrections, and readings will be documented in the field notebook per SOP F303.

**Laboratory Instruments**

All instrumentation used to perform chemical measurements must be properly calibrated to obtain valid and usable results. Calibration procedures for a specific laboratory instrument will consist of initial calibration (generally three to five points), initial calibration verification (inorganic methods only), and continuing calibration verification. In all cases, an independently prepared standard (i.e., from a second source or a different lot number from the primary source) will be used as a calibration verification solution or as the LCS/MS spiking mix.

All standards used to calibrate analytical instruments must be obtained from the National Institute of Standards and Technology (NIST) or through a reliable commercial supplier with a proven record for quality standards. All commercially supplied standards will be traceable to NIST reference standards, where possible, and appropriate documentation will be obtained from the supplier. In cases where documentation is not available, the laboratory will analyze the standard and compare the results with U.S. EPA-known or previous NIST-traceable standard.

Calibration procedures, frequency requirements, acceptance criteria, and conditions that require recalibration are described for each analytical procedure in the applicable laboratory SOPs included in Appendix D of this QAPP and Table 2-5.

## 2.7 Inspection/Acceptance Requirements for Supplies and Consumables

Evaluation and selection of suppliers and vendors is done, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short-term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff. Chemical reagents, solvents, glassware and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased.

## 2.8 Data Acquisition Requirements (Non-direct measurements)

This Section of the QAPP describes sources of previously collected data and other information that will be used to make project decisions. Since only Phase I's with no sampling and analysis have been performed to date at these sites, no data exists. Decision making for this project will be based solely on the samples taken during the field activity. The analytical results will be compared to the IDEM VRP-based screening criterias. Further details on this comparison process is provided in Section 1 of the QAPP.

As preparation of the Phase II investigation the following reports were reviewed:

- Phase I Environmental Site Assessment, Report Section 1, J-Pit Redevelopment Sites, Gary, Indiana, July 2002, Environmental Design International

- Phase I Environmental Site Assessment, Report Section 2, J-Pit Redevelopment Sites, Gary, Indiana, July 2002, Environmental Design International

- Phase I Environmental Site Assessment, Report Section 3, J-Pit Redevelopment Sites, Gary, Indiana, July 2002, Environmental Design International

- Phase I Environmental Site Assessment, Report Section 4, J-Pit Redevelopment Sites, Gary, Indiana, July 2002, Environmental Design International

- Phase I Environmental Site Assessment, Report Section 5, J-Pit Redevelopment Sites, Gary, Indiana, July 2002, Environmental Design International

- Hydrogeologic Report, from the Permit Application Glenwood Ridge, Restricted Wastes Disposal Facility, Lake County, Indiana, Volume 2 of 2. March, Rust Environmental.

## 2.9 Data Management

All data generated through field activities, or by the laboratory operation, will be reduced and validated prior to reporting. No data will be disseminated by the laboratory until it has been subjected to the procedures summarized below.

Field data will be transcribed directly from the instrument into the field logbook. If errors are made, results will be legibly crossed out, initiated and dated by the person recording the data, and corrected in a space adjacent to the original entry. Log books will be periodically reviewed by the FTL to ensure that records are complete, accurate, and legible.

Data at the laboratory is automatically entered into the LIMS from the instruments. In instances where instruments do not provide a "data dump" into the LIMS, the information is manually entered. Various checks are performed by the laboratory to ensure the integrity of the data. The laboratory will provide the analytical results electronically to Baker.

Baker's data management group will receive the analytical data from the laboratory electronically. Baker will check at a minimum ten percent of the electronic deliverable against the hard copy reports provided by the laboratory. Transcription errors will be documented and changed in the electronic

version of the data. Baker's data management group will incorporate data validation qualifiers into the electronic version of the data prior to use in the assessment phase of the project.

Upon successful completion of the data validation process for both the on-site and off-site data, the analytical results with validation qualification will be tabulated and stored on computer disks. The results will be presented in a user-friendly tabular format. The presentation will facilitate the review and use of the data to meet the project objectives.

## 2.10 Final Document

An outline of proposed Phase II Investigation report is as follows:

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# SECTION 3.0

# 3.0 ASSESSMENT/OVERSIGHT

## 3.1 Assessment and Response Actions

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the SAP and QAPP. These audits provide an indication of the data quality generated by the investigation. The audits of field and laboratory activities include two independent parts: internal and external audits.

### Field Performance and System Audits

#### Internal Field Audits

Internal Field Audit Responsibilities

The role of the QA Manager, including Quality Assurance, is described in Section 3.0. His/her responsibilities will include internal audits of field activities including sampling and field measurements.

Internal Field Audit Frequency

These audits will evaluate compliance with established procedures. Internal field audits will be conducted at least once at the beginning of sampling and analysis activities, so that problems, if any, and their solutions can be identified early. One additional audit may be conducted during subsequent sample collection activities.

Internal Field Audit Procedures

The audits will include examination of field sampling records, field instrument operating records, sample collection, handling and packaging in compliance with the established procedures, maintenance of QA procedures, chain-of-custody, etc. Follow-up audits will be conducted as required to correct deficiencies. The audits will involve review of field measurement records, instrumentation calibration records, and sample documentation.

**External Field Audits**

External Field Audit Responsibilities

Sima is a contracted laboratory for IDEM and is a nelap accredited laboratory under the Illinois EPA accreditation program (Appendix D)

External Field Audit Frequency

External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the USEPA or IDEM.

Overview of the External Field Audit Process

External field audits will be conducted according to the field activity information presented in the QAPP.

Performance and system audits for sampling and analysis operations consist of on-site review of field and laboratory quality assurance systems and on-site review of equipment for sampling, calibration, and measurement.

**Laboratory Performance and Systems Audits**

A performance audit is a check by Quality Assurance personnel of the major analyses conducted in the laboratory. This audit consists of, but is not limited to:

- A blind check sample for each department.
- Determination that proper quality control and corrective action procedures were employed in analysis of the blind sample
- Oversight of all analysts performing their major job function.
- Insure proper technique

A report of deficiencies determined in the performance audit will be submitted to laboratory management and immediate corrective action procedures will be adopted.

System audits are checks performed by quality assurance personnel of the entire laboratory operations. This audit consists of cradle to grave tracking of randomly selected samples through the entire analysis process. The audit contents will consist of, but not be limited to:

- Sample receipt practices, chain of custody
- Analysis
- Proper quality control
- Proper corrective-action documentation
- Records keeping and data storage
- Instrument prevention maintenance
- Review of final report

A report of deficiencies determined in the system audit will be submitted to management and immediate corrective action procedures will be implemented. The report becomes part of the permanent laboratory record.

Internal Laboratory Audits

Performance and system audits are designed to assess the quality of the total laboratory operation and to assure adherence to the quality control procedures specified in this QAPP.

Internal Lab Audit Responsibilities

The internal laboratory audit will be conducted by laboratory QA officer.

Internal Lab Audit Frequency

Both performance and system audits should be conducted semi-annually.

Internal Lab Audit Procedures

The internal lab system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of- custody procedures, sample preparation and analysis, instrument operating records, etc. The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The laboratory QA Officer will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance.

External Laboratory Audits

The lab is part of NELAP nelaps certification process, a copy of their certification from the state of Illinois is provided in Appendix D.

External Lab Audit Responsibilities

External field audits may be conducted any time during the field operations.

External Lab Audit Frequency

The frequency of the external audit will be at the discretion of the auditing agency (i.e., USEPA and IDEM). An external lab audit may be conducted at least once prior to the initiation of the sampling and analysis activities.

Overview of the External Lab Audit Process

External lab audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis.

## Corrective Action

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of quality control performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment. All corrective action proposed and implemented will be documented in a quality assurance report to management. Corrective action should only be implemented after approval by the Project Manager, or his designee, the Field Team Leader. If immediate corrective action is required, approvals secured by telephone from the Project Manager should be documented in an additional memorandum.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Project Manager or his designee if the problem occurs in the field, or the laboratory Project Manager if the problem occurs in the laboratory. Information on these problems will be promptly communicated to Baker's Field Team Leader and/or Project Manager who will communicate this issue to the City of Gary. Implementation of corrective actions will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the QAPP or SAP will be identified and corrected in accordance with the QAPP.

Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings and approval by the Project Manager. Corrective actions will be defined by the auditor and implemented to the satisfaction of the Project Manager. If corrective actions are insufficient, work may be stopped by a stop-work order issued by the Baker Project Manager or City of Gary.

## Field Corrective Action

Technical staff and project field personnel will be responsible for reporting all suspected technical or QA nonconformance or suspected deficiencies of any activity or issued document by reporting the

situation to the Field Team Leader or his/her designee. The Field Team Leader will be responsible for assessing the suspected problem, consulting with the Baker Project Manager on the problem and anticipated change, and implementing the change. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the Baker Project Manager.

The Baker Project Manager will be responsible for informing the City of Gary of the nonconformance. The City of Gary Project Manager or designee will inform IDEM and/or USEPA Project Manager of the problem.

The Baker Project Manager will be responsible for ensuring that corrective action for nonconformance is initiated by:

- Evaluating all reported nonconformance.
- Controlling additional work on nonconforming items.
- Determining disposition or action to be taken.
- Maintaining a log of nonconformance.
- Reviewing nonconformance reports and corrective actions to be taken.
- Ensuring nonconformance reports are included in the final facility documentation in project files.

If appropriate, the Baker Project Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

Corrective action for field measurements may include:

- Repeating the measurement to check the error.
- Checking all proper adjustments for ambient conditions such as temperature.
- Checking the batteries.
- Checking the calibration.
- Re-calibration.
- Replacing the instrument or measurement device.
- Stopping work (if necessary).

All changes will be evaluated based on the potential to impact the quality of the data. The Baker Project Manager has ultimate responsibility for all site activities. In this role, the Project Manager at times is required to adjust the site programs to accommodate site-specific needs. When it becomes necessary to modify a program, the responsible Field Team Leader notifies the Baker Project Manager of the anticipated change and implements the necessary changes. The Baker Project Manager or his designee must approve all changes verbally and/or in writing prior to field implementation by the Field Team Leader. The Project Manager will be notified when any field changes are made.

All problems and corrective actions will be documented in the field logbook by the Field Team Leader. No field team member will initiate corrective action without prior communication of findings through the proper channels. The action taken during the period of deviation will be evaluated in order to determine the significance of any departure from established program practices and action taken. If corrective actions are insufficient, work may be stopped by the Field Team Leader following instructions from the Project Manager or other designee.

Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the laboratory QA Manager to approve the implementation of corrective action. The submitted standard operating procedures (SOPs) specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, automatic reinjection/reanalysis when certain quality control criteria are not met, etc. It is the responsibility of the analyst and the laboratory QC Manager to develop and implement corrective action. It is the responsibility of the laboratory QC Manager and the Laboratory Manager to approve corrective actions and notify the Baker Project Manager of corrective actions taken. Any material modifications will be orally relayed by Baker Project Manager to the City of Gary Project Manager. The Cite of Gary Project Manager or designee will inform the IDEM or USEPA Project Manager who should state at the time of notification whether the corrective action is appropriate.

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy;
- Blanks contain target analytes above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;

- Deficiencies are detected by the QA Department during internal or external audits or from the results of performance evaluation samples; or

- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QC department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QC department.

These corrective actions are performed prior to release of the data from the laboratory. The corrective actions will be documented in both the laboratory's corrective action log (signed by analyst, section leader and quality control coordinator), and the narrative data report sent from the laboratory to the data validator. If corrective action dos not rectify the situation, the laboratory will contact the Baker Project Manager.

## 3.2 Reports to Management

### QUALITY ASSURANCE REPORT TO MANAGEMENT

The deliverables associated with the tasks identified in the Work plan will contain separate QA sections in which data quality information collected during the task is summarized. Those reports will be the responsibility of the Project Manager and will include the QA Officer report on the accuracy, precision, and completeness of the data as well as the results of the performance and system audits, and any corrective action needed or taken during the project.

## Contents of Project QA Report

The QA report will contain all results of field and laboratory audits, all information reflecting the achievement of specific data quality objectives, and a summary of corrective action that was implemented, and its immediate results on the project. The status of the project with respect to the Project Schedule included in the QAPP will be determined. Whenever necessary, updates on training provided, changes in key personnel, anticipated problems in the field or lab for the coming month that could bear on data quality along with proposed solutions, will be reported. Detailed references to QAPP modifications will also be highlighted. The QA report will be prepared in written, final format by the Project Manager or his designee.

In the event of an emergency, or in case it is essential to implement corrective action immediately, QA reports can be made by telephone to the appropriate individuals, as identified in the Project Organization or Corrective Action Sections of this QAPP. However, these events, and their resolution will be addressed thoroughly in the next issue of the monthly QA report.

## Frequency of QA Report

The QA Report will be incorporated into the final project report. The report will cover without interruption, the project through completion. The frequency of any emergency reports that must be delivered verbally cannot be estimated at the present time.

## Individuals Receiving/Reviewing QA Report

All individuals identified in the Project Organization chart will be notified about QA problems/issues.

# SECTION 4.0

# 4.0 DATA VALIDATION AND USABILTY

To properly utilize the results of the sampling investigation and laboratory analyses, the quality of the resulting field and analytical data must be assessed. Only data of appropriate quality should be used to produce conclusions about the Site. To produce a meaningful Phase II ESA, data must reflect the objectives listed in Section 1. In the event the objectives are not fulfilled, additional data may need to be required to reconcile the inadequacies. Consequently, data collected as part of this program needs to be verified and validated and its usability defined before producing any conclusions about the site from the data.

## 4.1 Data Verification and Validation Procedures

### 4.1.1 Field Verification

Field notes will be verified by the Baker Project Manager. Daily, the Baker Project Manager will review the field logs for completeness, accuracy and compatibility between sample locations and field samplers. He/She will sign and date the field log entry after review. Any required corrective action will be addressed with the field samplers prior to further work.

Chain-of-custody (COC) forms and shipping documentation will be reviewed by the Baker field samplers by comparing the information on the forms to the actual samples in the coolers.
Audit reports and associated corrective actions will be reviewed by the Baker Project Manager to ensure that appropriate corrective actions are being taken. He/She will sign and date the corrective action report after review.

**Field Data Reduction Procedures**

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Only direct read instrumentation will be employed in the field. The use of pH meters, thermometers, an HNu, and a probe to measure specific conductance will generate some measurements directly read from the meters following calibration per manufacturer's recommendations. Such data will be written into field log books immediately after measurements are taken. If errors are

made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, after the result forms required for this study are filled out, the forms will be proofed to determine whether any transcription errors have been made.

### 4.1.2 Laboratory Verification

The laboratory verification procedures ensure that data sets are complete, SOPs are followed, and documenting complete analytical events. Additionally, Sima evaluates each data set in accordance with the method performance criteria provided in Section 1. A brief description of the laboratory verification process is presented below.

Data Review

Sima Laboratory implements a two-tier review process. The first review is a technical review performed by the laboratory technician that conducted the analysis. This involves ensuring that the calculations are correct and that QC requirements were met. Quality control data (e.g. laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates) will be compared to the method acceptance criteria. The Data Review Form/QC Checklist is initiated by the Technician to document this review. Acceptable data are not ready for incorporation into the Laboratory Management Information System (LIMS). The Data Review Form/QC Checklist is now given to a peer knowledgeable of the analytical process, a Senior Technician, Unit Supervisor, QA/QC Director or the General Manager. A copy of the checklist is provided as Appendix D.

The second tier review is a verification of the review done by the analyst. This step is documented on the Data Review Form/QC Checklist. Data entered into the LIMS electronically may be reviewed electronically. Data entered into the LIMS manually must be reviewed using the raw data, where a representative number of calculations are verified and the quality control parameters are evaluated.

After this review, the data are now available for approval in the LIMS. If approved, data are logged into the project database format. Unacceptable data shall be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell

outside acceptance limits, and any other anomalous conditions encountered during sample analysis.. After approval, the data are reviewed by the laboratory Project Manager and released to the client.

Data Recording

Laboratory data recording procedures will be conducted in accordance with the following protocol: All raw analytical data will be recorded in numerically identified laboratory notebooks. These notebooks will be issued only by the Laboratory QA Manager. Data are recorded in this notebook along with other pertinent information, such as the sample identification number and the sample tag number. Other details will also be recorded in the lab notebook, such as the analytical method used (SOP Number), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook shall be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the Lab QA Manager takes place prior to final data reporting. (Records of notebook entry inspections are maintained by the Lab QA Manager.)

Data Reduction

The analytical SOPs provided in Appendix D provide the formulae used in data conversions (e.g., calculation of dry weight field sample concentrations) and the calculations used to quantitate the compound/element concentration from the instrument readouts. For this project, all solid sample results will be presented as dry weight; therefore, the data conversion will be conducted for each solid sample. In the process, the actual instrument printout provides information to calculate the analytes concentration. All calculations are checked by the laboratory section supervisor at the conclusion of each operating day. Errors are noted, corrections are made, but the original notations are crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis.

## 4.2 VALIDATION

Data Validation

Data validation procedures shall be performed for both field and laboratory operations as described below. Data validation will be performed by ECT CON

### 4.2.1 Procedures Used to Evaluate Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of field log books, on the part of field crew members. This task will be the responsibility of the Field Team Leader and the QA Manager.

### 4.2.2 Procedures to Validate Laboratory Data

Laboratory analytical data will be validated in accordance with current Data validation will be conducted in accordance with the USEPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review, February 1994, project SOPs, and professional judgement.

One hundred percent of the laboratory analytical data will be subjected to data validation to ensure that the data are of evidentiary quality. Validation of analytical data will be completed by an independent third party.

Data validators will review the chemical analytical data packages submitted by the laboratory. Analytical results will be validated versus the applicable analytical methods, the SOPs included in Appendix D of this QAPP, and the requirements of this QAPP. Validation of these data including the use of qualifying flags will conform to the National Functional Guidelines for Data Validation to the greatest extent practicable for non-CLP data. It should be noted that the analytical methods chosen for this project are from USEPA SW-846 "Test Methods for Evaluating Solid Waste". QC criteria used in these methods are often difference than that specified in the data validation guidelines. The criteria to

be used for this project are presented in Section 1.5 of this QAPP. The data validator will generate a report describing data limitations. The validation of off-site laboratory data will include the review of all QC information and raw data. QC supporting information will be reviewed to determine whether any data are outside established control limits, and if out-of-control data are discovered, appropriate corrective action will be determined. Any out-of-control data without appropriate corrective action will be cause to qualify the affected measurement data.

The data validation reports will be reviewed by the Baker QA Officer and the Baker Data Management group. The Baker Data Management group will compare the electronic data to the hard copy (e.g., Form 1) provided by the validator. Also, these individuals will incorporate the validator qualifiers into the electronic database. After review and incorporation into the database, the individual will sign and date the hard copy of the data provided by the validator.

## 4.3 Reconciliation with Data Quality Objectives

## 4.4 Data Reporting

Data reporting procedures shall be carried out for field and off-site laboratory operations as indicated below.

### Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities.

### Laboratory Data Reporting

The laboratory data packages will consist of all pertinent sample and project information, which will include the following (as applicable):

1. Case Narrative:

- Date of issuance
- Laboratory analysis performed
- Any deviations from intended analytical strategy
- Laboratory batch number
- Quality control procedures utilized and also references to the acceptance criteria
- Laboratory report contents
- Project name and number
- Condition of samples 'as-received'
- Discussion of whether or not sample holding times were met
- Discussion of technical problems or other observations which may have created analytical difficulties
- Discussion of any laboratory quality control checks which failed to meet project criteria
- Signature of the Laboratory QA Manager

2. Chemistry Data Package

- Case narrative for each analyzed batch of samples
- Summary page indicating dates of analyses for samples and laboratory quality control checks
- Cross referencing of laboratory sample to project sample identification numbers
- Data qualifiers to be used should be adequately described
- Sample preparation and analyses for samples
- Sample results
- Raw data for sample results and laboratory quality control samples
- Results of (dated) initial and continuing calibration checks, and GC/MS tuning results

- Matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results
- Labeled (and dated) chromatograms/spectra of sample results and laboratory quality control checks

The data package for the off-site laboratory will be a "CLP-like " data package consisting of all the information presented in a CLP data package and with QC summaries on CLP-like forms.

All data generated for the Site by the laboratory will be computerized in a format organized to facilitate data review and evaluation. The computerized data set will include the data flags provided by the laboratory in accordance with their SOPs. The laboratory-provided data flags will include such items as: estimated concentration due to poor spike recovery and concentration of chemical also found in laboratory bank.

For Sima, the hard copy final data package shall be delivered within 20 working days of the receipt of the last sample set. The electronic data report for each sample set shall be delivered within 20 working days of sample receipt.

The Data Validator comments will indicate that the data are: 1) usable as a quantitative concentration, 2) usable with caution as an estimated concentration, or 3) unusable due to out-of-control QC results. The validator qualifying flags will be incorporated into the electronic database. The final data set used to draw conclusions about the site will contain only the data validator qualifiers.

Baker's data management group will receive the analytical data from the laboratory electronically. Baker will check at a minimum ten percent of the electronic deliverable against the hard copy reports provided by the laboratory. Transcription errors will be documented and changed in the electronic version of the data. Baker's data management group will incorporate data validation qualifiers into the electronic version of the data prior to use in the assessment phase of the project.

Upon successful completion of the data validation process for both the on-site and off-site data, the analytical results with validation qualification will be tabulated and stored on computer disks. The results will be presented in a user-friendly tabular format. The presentation will facilitate the review and use of the data to meet the project objectives.

## 4.5 Usability/Reconciliation with Data Quality Objectives

The purpose of this section is to indicate the methods by which it will be ensured that the data collected for this investigation coincides with the project DQOs. The data assessment will be conducted to evaluate if the analytical data quality is in compliance with the QC objectives listed in Table 2-1 of this QAPP. Additionally, the data will be reviewed for indications of interferences to results caused by sample matrices, cross contamination during sampling, cross contamination in the laboratory, and sample preservation and storage anomalies.

Section 1 of this QAPP presents the precision, accuracy, representativeness, completeness and sensitivity DQOs for this project. Also presented in Section 1 are the calculations to be used to assess adherence to the DQOs for these QC items.

A review of the assessment criteria for these objectives is provided below.

Accuracy Assessment

The accuracy of field measurements will be assessed by adhering to proper field sampling and handling procedures as well as field instrument calibration procedures. Field measurement accuracy will be qualitatively reviewed and assessed.

In order to assure the accuracy of the analytical procedures, the laboratory uses primarily blank spikes (Laboratory control samples), system monitoring compounds and matrix spike samples. In general, a known amount of a spiking agent is added to a sample. The resulting concentration of the spiked compound divided by the true concentration is the percent recovery. Acceptable percent recovery limits are provided in Table 1-2.

## Precision Assessment

The field precision will be evaluated by calculating the RPDs for duplicate samples. Results of the field duplicate samples will be compared to the RPD goal of 50% for soil and 20% for water samples.

Matrix spiked samples are prepared by dividing the sample into equal aliquots, and then spiking each of the aliquots with a known amount of analyte. The matrix spiked duplicate samples will be then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The relative percent difference (RPD) between the spike and duplicate spike will be calculated and plotted. For this project, acceptable RPD for field duplicate samples is $\leq$50% for solid samples and $\leq$ 20% for aqueous samples. Table 1-2 presents acceptable RPD limits for analytical duplicate samples.

## Completeness Assessment

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. For this project, the completeness goal for the laboratory is 95% and for field measurements 90%.

## Sensitivity

The achievement of method detection limits depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure the data quality through constant instrument performance. The instrument sensitivity will be monitored through the analysis of method blank, calibration check sample, laboratory control samples, etc.

Laboratory adherence to these DQOs (precision, accuracy and sensitivity) will be assessed in the data validation process described in Section 4 of this QAPP including a review of QC items listed above. The analytical data will be validated in accordance with the National Functional Data Validation Guidance for Organics and Inorganics. Deviations in the QC criteria will be reported via data

qualifying flags, when applicable, or explained in the case narrative if qualification of the data is not deemed necessary. Other issues to be noted in the validation report are as follows:

- Deviations, if any, from the laboratory analytical SOPs
- Deviations, if any, from the data validation procedures
- Identification and explanation of elevated reporting limits

Field adherence to these DQOs (precision, accuracy, and representativeness) will be addressed in the Data Assessment Report. Deviations from the DQOs listed in Section 1 will be explained in the Data Assessment Report. Any uncertainty (e.g., biased high or biased low) associated with the data based on these deviations will be described and incorporated into the decision making process. Field duplicate samples will be used to assess precision. Trip and equipment blanks will be used to assess field accuracy. To assess the impact of trip and equipment blanks on field samples a comparison between the constituents detected in the blank samples and associated field samples will be conducted. Constituents detected in the field sample at a concentration less than five times (ten times for typical laboratory contaminants - acetone and methylene chloride), the concentration of the same constituent detected in the trip blank and/or equipment blank will be attributed to field cross-contamination. In this case, the presence of the constituent will not be associate with site conditions and will not be used in the decision making process. Other usability issues to be reviewed are as follows:

- Deviations, if any, from the field sampling SOPs
- Deviations, if any, from the sampling locations and sample number

Completeness will be assessed based on the number of usable data points from the overall project data points. Completeness will be calculated on both an area specific and overall project basis. For this project, a completeness goal of 95% or greater is required for the off-site laboratory and 95% for field measurements. Data points identified as unusable (i.e., data qualified as "R") are not included in the completeness calculation. The equation used to compute completeness is presented in Section 1 of this QAPP. Information on data completeness will be presented in the Data Assessment Report. If the completeness DQO is not meet for an area or the overall project, further evaluation of the site will be

required prior to final closure of the area. Further evaluation may or may not include intrusive measures.

Only data generated in association with QC results meeting these objectives and deemed usable by data validation will be considered usable for decision-making purposes. Limitations on the data will be reported via validation qualifying flags on the data, when applicable. For situations in which validation qualifying flags are not appropriate, a description of the limitation and the impact on the use of the data will be presented in the Data Assessment Report and taken into consideration in the decision making process.

As stated in Section 1 of the QAPP, the following evaluation will be performed with the analytical results:

The analytical results will be directly compared to the IDEM VRP-based screening criteria. This comparison will be performed in a tabular format. The table will contain the VRP-based screening criteria and the analytical results. Analytical results that exceed the screening criteria will be highlighted on the table. If a discrete sample result exceeds the screening criteria, further evaluation of the site will be required prior to final closure of the area. Further evaluation may or may not include intrusive measures.

# TABLES

TABLE 1-1
Distribution List and Document Control Numbers

| Company | Contact | Title | Phone | Fax | Email | Document Control Number |
|---|---|---|---|---|---|---|
| USEPA | Jan Pels | Brownfields Project Manager | (312) 886-3009 | | jan@epa.gov | J-Pit-1,2 |
| IDEM | Ruth Williams | Project Manager | (317) 233-4623 | | rwilliam@dem.state.in.us | J-Pit-3,4 |
| City of Gary | Mary Mulligan | Project Manager | (219) 882-3000 | (219) 882-3000 | mmulligan@ci.gary.in.us | J-Pit-5 |
| | Dorreen Carey | Project Manager | (219) 882-3000 | (219) 882-3000 | dcarey@ci.gary.in.us | J-Pit-6 |
| Baker Environmental, Inc | Rick Spitaler | Geologist | (219) 736-0263<br>(219) 808-8659 | (219) 755-0233 | rspitaler@mbakercorp.com | J-Pit-7 |
| | Jim Peyton | Sr. Geologist | (219) 736-0263<br>(219) 808-8657 | (219) 755-0233 | jpeyton@mbakercorp.com | J-Pit-7 |
| | Kurt Weiss | Environmental Scientist | (219) 736-0263 | (219) 755-0233 | kweiss@mbakercorp.com | J-Pit-7 |
| V3Consultants | R. Damon Lee | Director of Environmental Redevelopment | (630) 724-9200 ext.153 | (630) 724-9202 | Dlee@v3consultants.com | J-Pit-8 |
| SimaLab | Michelle Dilley | Laboratory Project Manager | (219) 769-8378 | | | J-Pit-9 |
| | Jeff Loewe | QA Manager | (219) 769-8378 | | | J-Pit-9 |

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil [1] Residential | | | | | ✱ Groundwater[2] Residential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level (mg/kg) | PQL[3] (mg/kg) | Accuracy [4] LCL % | Accuracy [4] UCL % | Precision [4] RPD % | Screening Level (mg/l) | PQL[3] (mg/l) | Accuracy [5] LCL % | Accuracy [5] UCL % | Precision [5] RPD % |
| **Semivolatile Organic Compounds** | | | | | | | | | | |
| naphthalene | 1,761.785 | 0.660 | NA | NA | NA | 1.21600 | 0.01000 | NA | NA | NA |
| acenaphthylene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA | NA |
| acenaphthene | 10,000.000 | 0.660 | 17.1 | 89.1 | 24.5 | 1.82400 | 0.01000 | 14.5 | 96.6 | 41.3 |
| fluorene | 8,838.641 | 0.660 | 29 | 156 | 46 | 1.21600 | 0.01000 | NA | NA | NA |
| phenanthrene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA | NA |
| anthracene | 10,000.000 | 0.660 | NA | NA | NA | 9.12000 | 0.01000 | NA | NA | NA |
| fluoranthene | 2,305.040 | 0.660 | NA | NA | NA | 0.24320 | 0.01000 | NA | NA | NA |
| pyrene | 10,000.000 | 0.660 | 5 | 116 | 21.4 | 0.91200 | 0.01000 | 14.3 | 107 | 37.7 |
| benzo(a)anthracene | 103.881 | 0.660 | NA | NA | NA | 0.00010 | 0.01000 | NA | NA | NA |
| chrysene | 379.273 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA | NA |
| benzo(b)fluoranthene | 354.977 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA | NA |
| benzo(k)fluoranthene | 501.638 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA | NA |
| benzo(a)pyrene | 69.849 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA | NA |
| indeno(1,2,3-cd)pyrene | 629.166 | 0.660 | NA | NA | NA | 0.00040 | 0.01000 | NA | NA | NA |
| dibenzo(a,h)anthracene | 69.863 | 0.660 | NA | NA | NA | 0.00030 | 0.01000 | NA | NA | NA |
| benzo(g,h,i)perylene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA | NA |
| 3,3'-dichlorobenzidine | 12.865 | 1.300 | NA | NA | NA | 0.02000 | 0.02000 | NA | NA | NA |
| n-nitroso-di-n-propylamine | 0.660 | 0.660 | 9.25 | 109 | 34 | 0.01000 | 0.01000 | 6.65 | 116 | 42.7 |
| bis(2-chloroisopropyl)ether | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| 4-chloroaniline | 186.921 | 1.300 | NA | NA | NA | 0.12160 | 0.02000 | NA | NA | NA |
| 2-chloronaphthalene | 10,000.000 | 0.660 | NA | NA | NA | 2.43200 | 0.01000 | NA | NA | NA |
| 2,4-dinitrotoluene[4] | 6.535 | 0.660 | 12.5 | 83.4 | 29.7 | 0.06080 | 0.01000 | 18.7 | 96.2 | 36.5 |
| hexachlorobutadiene | 6.777 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| hexachloroethane | 1.153 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| isophorone | 1.433 | 0.660 | NA | NA | NA | 0.08947 | 0.01000 | NA | NA | NA |
| benzyl alcohol | 728.618 | 1.300 | NA | NA | NA | 9.12000 | 0.02000 | NA | NA | NA |
| bis(2-chloroethyl)ether | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| nitrobenzene | 0.660 | 0.660 | NA | NA | NA | 0.01520 | 0.01000 | NA | NA | NA |
| 1,2-dichlorobenzene | 2,524.230 | 0.660 | NA | NA | NA | 0.60000 | 0.01000 | NA | NA | NA |
| 1,3-dichlorobenzene | NA | 0.660 | NA | NA | NA | 0.60000 | 0.01000 | NA | NA | NA |
| 1,4-dichlorobenzene | 0.897 | 0.660 | 5 | 93.1 | 31.1 | 0.07500 | 0.01000 | 12.1 | 93.1 | 39.7 |
| 1,2,4-trichlorobenzene | 235.033 | 0.660 | 5 | 86.2 | 34.6 | 0.07000 | 0.01000 | 6.56 | 93.1 | 44.8 |
| hexachlorobenzene[4] | 101.564 | 0.660 | NA | NA | NA | 0.00100 | 0.01000 | NA | NA | NA |
| hexachlorocyclopentadiene | 2.891 | 0.660 | NA | NA | NA | 0.05000 | 0.01000 | NA | NA | NA |
| n-nitrosodiphenylamine | 3.177 | 0.660 | NA | NA | NA | 0.01735 | 0.01000 | NA | NA | NA |
| benzoic acid | 10,000.000 | 3.300 | NA | NA | NA | 121.60000 | 0.05000 | NA | NA | NA |
| 2-nitroaniline | 3.300 | 3.300 | NA | NA | NA | 0.05000 | 0.05000 | NA | NA | NA |

✱ Groundwater samples will be analyzed for PAHs using Method 8310 (see Appendix D for PQLs) and SVOCs using Method 8270.

**TABLE 1-2**
**DATA QUALITY OBJECTIVES**

| Constituent | Subsurface Soil [1] Residential | | | | | Groundwater [2] Residential | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Screening Level (mg/kg) | PQL [3] (mg/kg) | Accuracy [4] LCL % | Accuracy [4] UCL % | Precision [4] RPD % | Screening Level (mg/l) | PQL [3] (mg/l) | Accuracy [5] LCL % | Accuracy [5] UCL % | Precision [5] RPD % |
| phenol | 110.173 | 0.660 | 10.8 | 87.8 | 31.4 | 3.64800 | 0.01000 | 5 | 70.4 | 55 |
| 2-methylphenol | 62.871 | 0.660 | NA | NA | NA | 1.52000 | 0.01000 | NA | NA | NA |
| 3-methylphenol | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA | NA |
| 4-methylphenol | 71.452 | 0.660 | NA | NA | NA | 1.52000 | 0.01000 | NA | NA | NA |
| 2-chlorophenol | 1.945 | 0.660 | 19.6 | 76.5 | 32 | 0.15200 | 0.01000 | 5 | 106 | 44.3 |
| 2,4-dichlorophenol[4] | 2.528 | 0.660 | NA | NA | NA | 0.09120 | 0.01000 | NA | NA | NA |
| 2,4,5-trichlorophenol | 921.059 | 0.660 | NA | NA | NA | 3.04000 | 0.01000 | NA | NA | NA |
| 2,4,6-trichlorophenol[4] | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA | NA |
| pentachlorophenol | 24.947 | 3.300 | 5 | 91.3 | 12.2 | 0.00100 | 0.05000 | 5 | 135 | 29.2 |
| 2,4-dinitrophenol | 3.300 | 3.300 | NA | NA | NA | 0.06080 | 0.05000 | NA | NA | NA |
| bis(2-ethylhexyl)phthalate[4] | 16.427 | 0.660 | NA | NA | NA | 0.00600 | 0.01000 | NA | NA | NA |
| butylbenzylphthalate | 10,000.000 | 0.660 | NA | NA | NA | 0.10000 | 0.01000 | NA | NA | NA |
| di-n-butylphthalate[4] | 1,034.967 | 0.660 | NA | NA | NA | 0.60800 | 0.01000 | NA | NA | NA |
| diethylphthalate | 10,000.000 | 0.660 | NA | NA | NA | 24.32000 | 0.01000 | NA | NA | NA |
| de methyl phthalate | 10,000.000 | 0.660 | NA | NA | NA | 304.00000 | 0.01000 | NA | NA | NA |
| di-n-octyl phthalate | 2,318.850 | 0.660 | NA | NA | NA | 0.60800 | 0.01000 | NA | NA | NA |
| **Volatile Organic Compounds** | | | | | | | | | | |
| benzene | 0.059 | 0.005 | 54.3 | 134 | 32 | 0.00500 | 0.00500 | 60.6 | 130 | 23.7 |
| toluene | 278.926 | 0.005 | 45.3 | 147 | 44.7 | 1.00000 | 0.00500 | 69.7 | 130 | 25.4 |
| ethylbenzene | 834.372 | 0.005 | 33 | 161 | 70.9 | 0.70000 | 0.00500 | 60.6 | 144 | 25.2 |
| xylenes | 1,000.000 | 0.005 | NA | NA | NA | 10.00000 | 0.00500 | NA | NA | NA |
| vinyl chloride | 0.129 | 0.010 | 21.5 | 174 | 49.3 | 0.00200 | 0.01000 | 33 | 151 | 31.2 |
| chloroethane | 1,000.000 | 0.010 | 0 | 439 | 45 | 23.16075 | 0.01000 | 0 | 357 | 29.3 |
| 1,1-dichloroethylene | 0.084 | 0.005 | 40.5 | 140 | 36.4 | 0.00700 | 0.00500 | 50.1 | 125 | 31 |
| 1,1-dichloroethane | 40.074 | 0.005 | 60.5 | 134 | 24.1 | 0.64000 | 0.00500 | 68 | 119 | 14.2 |
| 1,2-dichloroethylene (cis) | 17.140 | 0.005 | NA | NA | NA | 0.07000 | 0.00500 | NA | NA | NA |
| 1,2-dichloroethane | 0.025 | 0.005 | 55.9 | 149 | 41.2 | 0.00500 | 0.00500 | 67.7 | 133 | 23.3 |
| trichloroethylene | 0.076 | 0.005 | 41.8 | 145 | 35.3 | 0.00500 | 0.00500 | 48.6 | 139 | 26.9 |
| 1,1,1-trichloroethane | 229.642 | 0.005 | 51.5 | 149 | 24.4 | 0.20000 | 0.00500 | 63 | 136 | 29 |
| 1,1,2-trichloroethane | 0.035 | 0.005 | 49.2 | 147 | 34.4 | 0.00500 | 0.00500 | 40.6 | 165 | 25.9 |
| tetrachloroethylene | 0.227 | 0.005 | 51.8 | 139 | 29.1 | 0.00500 | 0.00500 | 60.1 | 144 | 29.7 |
| 1,1,1,2-tetrachloroethane | 0.076 | 0.005 | NA | NA | NA | 0.00500 | 0.00500 | NA | NA | NA |
| 1,1,2,2-tetrachloroethane | 0.044 | 0.005 | 24.9 | 201 | 85.2 | 0.00500 | 0.00500 | 46.8 | 168 | 22.3 |
| chloroform | 2.082 | 0.005 | 59.8 | 126 | 22.1 | 0.10000 | 0.00500 | 71.2 | 120 | 25 |
| acetone | 22.793 | 0.100 | NA | NA | NA | 3.04000 | 0.10000 | NA | NA | NA |
| 4-methyl-2-pentanone | 68.147 | 0.050 | NA | NA | NA | 1.52000 | 0.05000 | NA | NA | NA |
| methyl ethyl ketone | 11.620 | 0.100 | NA | NA | NA | 0.91772 | 0.10000 | NA | NA | NA |

Table 1-2 - DQOs

| Constituent | Subsurface | | | | | roundwater(2 | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Screening | PQL(3) | Accuracy (4) | | Precision (4) | Screening | PQL(3) | Accuracy (5) | |
| | | | LCL | UCL | RPD | | | LCL | UCL |
| | (mg/kg) | (mg/kg) | % | % | % | (mg/l) | (mg/l) | % | % |
| **Semivolatile Organic Compounds** | | | | | | | | | |
| naphthalene | 1,761.785 | 0.660 | NA | NA | NA | 1.21600 | 0.01000 | NA | NA |
| acenaphthylene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA |
| acenaphthene | 10,000.000 | 0.660 | 17.1 | 89.1 | 24.5 | 1.82400 | 0.01000 | 14.5 | 96.6 |
| fluorene | 8,838.641 | 0.660 | 29 | 156 | 46 | 1.21600 | 0.01000 | NA | NA |
| phenanthrene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA |
| anthracene | 10,000.000 | 0.660 | NA | NA | NA | 9.12000 | 0.01000 | NA | NA |
| fluoranthene | 2,305.040 | 0.660 | NA | NA | NA | 0.24320 | 0.01000 | NA | NA |
| pyrene | 10,000.000 | 0.660 | 5 | 116 | 21.4 | 0.91200 | 0.01000 | 14.3 | 107 |
| benzo(a)anthracene | 103.881 | 0.660 | NA | NA | NA | 0.00010 | 0.01000 | NA | NA |
| chrysene | 379.273 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA |
| benzo(b)fluoranthene | 354.977 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA |
| benzo(k)fluoranthene | 501.638 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA |
| benzo(a)pyrene | 69.849 | 0.660 | NA | NA | NA | 0.00020 | 0.01000 | NA | NA |
| indeno(1,2,3-cd)pyrene | 629.166 | 0.660 | NA | NA | NA | 0.00040 | 0.01000 | NA | NA |
| dibenzo(a,h)anthracene | 69.863 | 0.660 | NA | NA | NA | 0.00030 | 0.01000 | NA | NA |
| benzo(g,h,i)perylene | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA |
| 3,3'-dichlorobenzidine | 12.865 | 1.300 | NA | NA | NA | 0.02000 | 0.02000 | NA | NA |
| n-nitroso-di-n-propylamine | 0.660 | 0.660 | 9.25 | 109 | 34 | 0.01000 | 0.01000 | 6.65 | 116 |
| bis(2-chloroisopropyl)ether | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA |
| 4-chloroaniline | 186.921 | 1.300 | NA | NA | NA | 0.12160 | 0.02000 | NA | NA |
| 2-chloronaphthalene | 10,000.000 | 0.660 | NA | NA | NA | 2.43200 | 0.01000 | NA | NA |
| 2,4-dinitrotoluene4 | 6.535 | 0.660 | 12.5 | 83.4 | 29.7 | 0.06080 | 0.01000 | 18.7 | 96.2 |
| hexachlorobutadiene | 6.777 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA |
| hexachloroethane | 1.153 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA |
| isophorone | 1.433 | 0.660 | NA | NA | NA | 0.08947 | 0.01000 | NA | NA |
| benzyl alcohol | 728.618 | 1.300 | NA | NA | NA | 9.12000 | 0.02000 | NA | NA |

| [redacted] |
| --- |
| |
| Precision (5) |
| RPD % |
| |
| NA |
| NA |
| 41.3 |
| NA |
| NA |
| NA |
| NA |
| 37.7 |
| NA |
| NA |
| NA |
| NA |
| NA |
| NA |
| NA |
| NA |
| NA |
| 42.7 |
| NA |
| NA |
| NA |
| 36.5 |
| NA |
| NA |
| NA |
| NA |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| bis(2-chloroethyl)ether | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA |
| nitrobenzene | 0.660 | 0.660 | NA | NA | NA | 0.01520 | 0.01000 | NA | NA |
| 1,2-dichlorobenzene | 2,524.230 | 0.660 | NA | NA | NA | 0.60000 | 0.01000 | NA | NA |
| 1,3-dichlorobenzene | NA | 0.660 | NA | NA | NA | 0.60000 | 0.01000 | NA | NA |
| 1,4-dichlorobenzene | 0.897 | 0.660 | 5 | 93.1 | 31.1 | 0.07500 | 0.01000 | 12.1 | 93.1 |
| 1,2,4-trichlorobenzene | 235.033 | 0.660 | 5 | 86.2 | 34.6 | 0.07000 | 0.01000 | 6.56 | 93.1 |
| hexachlorobenzene4 | 101.564 | 0.660 | NA | NA | NA | 0.00100 | 0.01000 | NA | NA |
| hexachlorocyclopentadiene | 2.891 | 0.660 | NA | NA | NA | 0.05000 | 0.01000 | NA | NA |
| n-nitrosodiphenylamine | 3.177 | 0.660 | NA | NA | NA | 0.01735 | 0.01000 | NA | NA |
| benzoic acid | 10,000.000 | 3.300 | NA | NA | NA | 121.60000 | 0.05000 | NA | NA |
| 2-nitroaniline | 3.300 | 3.300 | NA | NA | NA | 0.05000 | 0.05000 | NA | NA |
| phenol | 110.173 | 0.660 | 10.8 | 87.8 | 31.4 | 3.64800 | 0.01000 | 5 | 70.4 |
| 2-methylphenol | 62.871 | 0.660 | NA | NA | NA | 1.52000 | 0.01000 | NA | NA |
| 3-methylphenol | NA | 0.660 | NA | NA | NA | NA | 0.01000 | NA | NA |
| 4-methylphenol | 71.452 | 0.660 | NA | NA | NA | 1.52000 | 0.01000 | NA | NA |
| 2-chlorophenol | 1.945 | 0.660 | 19.6 | 76.5 | 32 | 0.15200 | 0.01000 | 5 | 106 |
| 2,4-dichlorophenol4 | 2.528 | 0.660 | NA | NA | NA | 0.09120 | 0.01000 | NA | NA |
| 2,4,5-trichlorophenol | 921.059 | 0.660 | NA | NA | NA | 3.04000 | 0.01000 | NA | NA |
| 2,4,6-trichlorophenol4 | 0.660 | 0.660 | NA | NA | NA | 0.01000 | 0.01000 | NA | NA |
| pentachlorophenol | 24.947 | 3.300 | 5 | 91.3 | 12.2 | 0.00100 | 0.05000 | 5 | 135 |
| 2,4-dinitrophenol | 3.300 | 3.300 | NA | NA | NA | 0.06080 | 0.05000 | NA | NA |
| bis(2-ethylhexyl)phthalate4 | 16.427 | 0.660 | NA | NA | NA | 0.00600 | 0.01000 | NA | NA |
| butylbenzylphthalate | 10,000.000 | 0.660 | NA | NA | NA | 0.10000 | 0.01000 | NA | NA |
| di-n-butylphthalate4 | 1,034.967 | 0.660 | NA | NA | NA | 0.60800 | 0.01000 | NA | NA |
| diethylphthalate | 10,000.000 | 0.660 | NA | NA | NA | 24.32000 | 0.01000 | NA | NA |
| de methyl phthalate | 10,000.000 | 0.660 | NA | NA | NA | 304.00000 | 0.01000 | NA | NA |
| di-n-octyl phthalate | 2,318.850 | 0.660 | NA | NA | NA | 0.60800 | 0.01000 | NA | NA |
| **Volatile Organic Compounds** | | | | | | | | | |
| benzene | 0.059 | 0.005 | 54.3 | 134 | 32 | 0.00500 | 0.00500 | 60.6 | 130 |
| toluene | 278.926 | 0.005 | 45.3 | 147 | 44.7 | 1.00000 | 0.00500 | 69.7 | 130 |
| ethylbenzene | 834.372 | 0.005 | 33 | 161 | 70.9 | 0.70000 | 0.00500 | 60.6 | 144 |
| xylenes | 1,000.000 | 0.005 | NA | NA | NA | 10.00000 | 0.00500 | NA | NA |
| vinyl chloride | 0.129 | 0.010 | 21.5 | 174 | 49.3 | 0.00200 | 0.01000 | 33 | 151 |
| chloroethane | 1,000.000 | 0.010 | 0 | 439 | 45 | 23.16075 | 0.01000 | 0 | 357 |
| 1,1-dichloroethylene | 0.084 | 0.005 | 40.5 | 140 | 36.4 | 0.00700 | 0.00500 | 50.1 | 125 |
| 1,1-dichloroethane | 40.074 | 0.005 | 60.5 | 134 | 24.1 | 0.64000 | 0.00500 | 68 | 119 |
| 1,2-dichloroethylene (cis) | 17.140 | 0.005 | NA | NA | NA | 0.07000 | 0.00500 | NA | NA |
| 1,2-dichloroethane | 0.025 | 0.005 | 55.9 | 149 | 41.2 | 0.00500 | 0.00500 | 67.7 | 133 |

| |
|---|
| NA |
| NA |
| NA |
| NA |
| 39.7 |
| 44.8 |
| NA |
| NA |
| NA |
| NA |
| NA |
| NA |
| 55 |
| NA |
| NA |
| NA |
| 44.3 |
| NA |
| NA |
| NA |
| 29.2 |
| NA |
| NA |
| NA |
| NA |
| NA |
| NA |
| NA |
| |
| 23.7 |
| 25.4 |
| 25.2 |
| NA |
| 31.2 |
| 29.3 |
| 31 |
| 14.2 |
| NA |
| 23.3 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| trichloroethylene | 0.076 | 0.005 | 41.8 | 145 | 35.3 | 0.00500 | 0.00500 | 48.6 | 139 |
| 1,1,1-trichloroethane | 229.642 | 0.005 | 51.5 | 149 | 24.4 | 0.20000 | 0.00500 | 63 | 136 |
| 1,1,2-trichloroethane | 0.035 | 0.005 | 49.2 | 147 | 34.4 | 0.00500 | 0.00500 | 40.6 | 165 |
| tetrachloroethylene | 0.227 | 0.005 | 51.8 | 139 | 29.1 | 0.00500 | 0.00500 | 60.1 | 144 |
| 1,1,1,2-tetrachloroethane | 0.076 | 0.005 | NA | NA | NA | 0.00500 | 0.00500 | NA | NA |
| 1,1,2,2-tetrachloroethane | 0.044 | 0.005 | 24.9 | 201 | 85.2 | 0.00500 | 0.00500 | 46.8 | 168 |
| chloroform | 2.082 | 0.005 | 59.8 | 126 | 22.1 | 0.10000 | 0.00500 | 71.2 | 120 |
| acetone | 22.793 | 0.100 | NA | NA | NA | 3.04000 | 0.10000 | NA | NA |
| 4-methyl-2-pentanone | 68.147 | 0.050 | NA | NA | NA | 1.52000 | 0.05000 | NA | NA |
| methyl ethyl ketone | 11.620 | 0.100 | NA | NA | NA | 0.91772 | 0.10000 | NA | NA |
| **Pesticides** | | | | | | | | | |
| Aldrin | 0.007 | 0.003 | 34 | 132 | 43 | 0.00004 | 0.00004 | 40 | 120 |
| gamma-BHC (Lindane) | 0.010 | 0.006 | 46 | 127 | 50 | 0.00020 | 0.00009 | 56 | 123 |
| chlordane | 4.512 | 0.009 | NA | NA | NA | 0.00200 | 0.00014 | NA | NA |
| DDD | 0.270 | 0.007 | NA | NA | NA | 0.00035 | 0.00011 | NA | NA |
| DDE | 0.450 | 0.003 | NA | NA | NA | 0.00025 | 0.00004 | NA | NA |
| DDT | 0.794 | 0.008 | NA | NA | NA | 0.00025 | 0.00012 | NA | NA |
| dieldrin | 0.003 | 0.001 | 31 | 134 | 38 | 0.00002 | 0.00002 | 52 | 126 |
| endosulfan sulfate | 2.007 | 0.044 | NA | NA | NA | 0.00152 | 0.00066 | NA | NA |
| endrin | 1.939 | 0.004 | 42 | 139 | 45 | 0.00200 | 0.00006 | 56 | 121 |
| heptachlor | 0.221 | 0.002 | 35 | 130 | 31 | 0.00040 | 0.00003 | 40 | 131 |
| heptachlor epoxide | 0.450 | 0.056 | NA | NA | NA | 0.00020 | 0.00083 | NA | NA |
| **PCBs** | | | | | | | | | |
| PCBs (Aroclor 1016) | 4.226 | 0.044 | 70 | 130 | NA | 0.00050 | 0.00065 | 70 | 130 |
| **Metals** | | | | | | | | | |
| lead | NA | 0.500 | 70 | 130 | 20 | NA | 0.00300 | 75 | 125 |
| cadmium | 730.000 | 0.500 | 70 | 130 | 20 | 0.00500 | 0.00500 | 75 | 125 |
| silver | 7,300.000 | 1.000 | 70 | 130 | 20 | 0.15200 | 0.01000 | 75 | 125 |
| mercury | 87.600 | 0.100 | 70 | 130 | 20 | 0.00200 | 0.00020 | 75 | 125 |
| chromium vi | 7,300.000 | 1.000 | 70 | 130 | 20 | 0.10000 | 0.01000 | 75 | 125 |
| chromium iii | 10,000.000 | 1.000 | 70 | 130 | 20 | 0.10000 | 0.01000 | 75 | 125 |
| barium | 10,000.000 | 20.000 | 70 | 130 | 20 | 2.00000 | 0.20000 | 75 | 125 |
| arsenic | 438.000 | 1.000 | 70 | 130 | 20 | 0.05000 | 0.01000 | 75 | 125 |
| antimony | 584.000 | 6.000 | 70 | 130 | 20 | 0.00600 | 0.06000 | 75 | 125 |
| beryllium | 118.605 | 0.500 | 70 | 130 | 20 | 0.00400 | 0.00500 | 75 | 125 |
| cyanide | 10,000.000 | 0.125 | 70 | 130 | 20 | 0.20000 | 0.01000 | 75 | 125 |
| nickel | 10,000.000 | 4.000 | 70 | 130 | 20 | 0.10000 | 0.04000 | 75 | 125 |
| selenium | 7,300.000 | 0.500 | 70 | 130 | 20 | 0.05000 | 0.00500 | 75 | 125 |

| |
|---|
| 26.9 |
| 29 |
| 25.9 |
| 29.7 |
| NA |
| 22.3 |
| 25 |
| NA |
| NA |
| NA |
| |
| 22 |
| 15 |
| NA |
| NA |
| NA |
| NA |
| 18 |
| NA |
| 21 |
| 20 |
| NA |
| |
| NA |
| |
| 20 |
| 20 |
| 20 |
| 20 |
| 20 |
| 20 |
| 20 |
| 20 |
| 20 |
| 20 |
| 20 |
| 20 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| vanadium | 10,000.000 | 5.000 | 70 | 130 | 20 | 0.21280 | 0.05000 | 75 | 125 |
| zinc | 10,000.000 | 2.000 | 70 | 130 | 20 | 9.12000 | 0.02000 | 75 | 125 |

Footnotes:

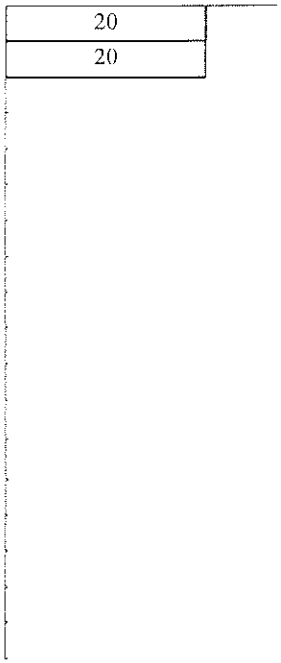
1- Tier II screening levels for subsurface

2- Tier II screening levels for groundwater

3- PQL - Practical Quantitation Limit, based on EPA SW-846, 1986 for GC/MS.

4- Compounds are not spiked in Method 8270.

NA-Not Applicable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20

**Table 1-3**
**Summary Table of Sampling Analysis Program**

| LOCATION | SOIL/SEDIMENT | | | SURFACE WATER/GROUNDWATER | | |
|---|---|---|---|---|---|---|
| | 8 RCRA METALS | VOLATILE ORGANIC COMPOUNDS (VOCs) | SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs) | 8 RCRA METALS | VOLATILE ORGANIC COMPOUNDS (VOCs) | SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs) |
| *Date Prepared: 7/02* | | | | | | |
| Section 1 | 2 | 7 | 7 | 4 | 4 | 4 |
| Section 2 | 2 | 8 | 8 | 4 | 4 | 4 |
| Section 3 | 2 | 8 | 8 | 4 | 4 | 4 |
| Section 4 | 2 | 7 | 7 | 2 | 2 | 2 |
| J-Pit Section 5 | 8 | 16 | 16 | 8 | 8 | 8 |
| Discretionary | 2 | 2 | 2 | 4 | 4 | 4 |
| QA/QC | 5 | 9 | 9 | 5 | 5 | 5 |
| Total | 23 | 57 | 57 | 31 | 31 | 31 |

**NOTE:**
Volatile Organic Compounds (VOCs) analyzed via USEPA SW-846 Method 8260B.
Semi-Volatile Organic Compounds (SVOCs) analyzed via USEPA SW-846 Method 8270A.
Metals (As, Ba, Cd, Cr, Pb, Se, and Ag) are analyzed via USEPA SW-846 Method 6010.
Hg is analyzed via USEPA Method SW-846 method 7471a for solid and 7470a for aqueous.

**Table 2-1**

**Sample Container, Preservation, Holding, and Volume Requirements**

| Matrix | Name | Containers | Preservation | Holding Times | Minimum Sample Volume or Weight Soil/Water |
|---|---|---|---|---|---|
| *Date Prepared: 7/02* | | | | | |
| **Soil** | Metals, *except* Cr(VI) | 8 oz. WMJ (s) | Cool, 4 ± 2 °C | Metals (except Hg): 6 months<br>Hg: 28 days | 1 g/100 mL Hg 5 g/100 mL ICP/GF AA |
| | Semi-Volatile Organics | 8 oz. WMJ (s) | Cool, 4 ± 2 °C | 14 days for (s), 40 days from collection | 30 g/ 1000 mL |
| | VOCs | 4 oz. WMJ (s) | Cool, 4 ± 2 °C | 14 days | 5 g/25 mL, No headspace: |
| **Water** | Metals, *except* Cr(VI) | 1 L HDPE (aq) | 5ml of 35% $HNO_3$ to pH<2(aq):<br>Cool, 4 ± 2 °C | Metals (except Hg): 6 months<br>Hg: 28 days | 1 g/100 mL Hg 5 g/100 mL ICP/GF AA |
| | Semi-Volatile Organics | 2-1 L AG with Teflon-lined cap (aq) | Cool, 4 ± 2 °C | 7 days until extraction (aq) | 30 g/ 1000 mL |
| | VOCs | 3-40 mL Borosilicate glass vial (aq) | 0.5 mL 36% HCl to pH<2 (aq):<br>Cool, 4 ± 2 °C. For chlorinated waters, add 25 mg ascorbic acid per vial prior to sampling. | 14 days for (aq) | 5 g/25 mL, No headspace: |

Notes:
HDPE = High Density Polyethylene bottle
aq = Aqueous Sample
AG = Amber, Boston round, Glass Bottle
s = Soil Sample
WMJ = Wide Mouth Paragon Flint Glass Jar
Metals include: Ar, Ba, Cd, Cr, Pb, Hg, Se and Ag

## TABLE 2-2

## QA OBJECTIVE FOR FIELD MEASUREMENTS

| Parameter | Measurement Instrument | Precision (1) | Accuracy (2) | Completeness |
|---|---|---|---|---|
| **Water** | | | | |
| Temperature | Temperature with the conductivity or pH meter | +/- 0.5 Deg. C | 0.05 Deg. C | 95% |
| pH | pH meter | +/- 0.1 pH units | 0.05 pH units | 95% |
| Specific conductance | Conductivity meter | +/- 25 umhos/cm | 10umhos/cm | 95% |
| Dissolved oxygen | DO meter | +/- 0.1 mg/L | 0.1 mg/L | 95% |
| Oxidation reduction potential | Redox meter | +/- 10 mV | 10 mV | 95% |

(1) Expressed as the acceptable deviation from the scale
(2) Expected based on equipment manufacturer's specification

## Table 2-3

## Field Calibration Procedures and Frequencies

| Instrument | Calibration Procedure | Frequency | Acceptance Criteria | Corrective Action Response |
|---|---|---|---|---|
| pH Meter | Calibrate to a standard solution of pH 7. | At beginning of a dailiy sampling, calibrate with both pH 7 and pH 4 or 10. | Acceptable when pH of standard is measured to withinn 0.05 pH units. | Check if pH meter battery and connections. |
| | Calibrate to a standard solution of pH 4 or 10 depending on applicable data range of expected pH readings. | Check calibration after measurement of a sample with any large variance pH. | | Check if pH probe is clean. Rinse with DO water. |
| | | | | Recalibrate with new pH standard solutions. |
| Conductivity Meter | Calibrate to a standard solution of of 1410 umho/cm$^2$. | At beginning of daily sampling, calibrate with standard. | Acceptable when conductivity of standard is measured to within 10 umho/cm$^2$. | Check conductivity meter battery and connections. |
| | | Check calibration after measurement of a sample with any large variance conductivity. | | Check if conductivity probe is clean. Rinse with DO water. |
| | | | | Recalibrate with new conductivity standard solution. |

## TABLE 2-4

## PREVENTATIVE MAINTENANCE FIELD EQUIPMENT

| INSTRUMENTS | MAINTENANCE PROCEDURES/SCHEDULE | SPARE PARTS IN STOCK |
|---|---|---|
| pH/Eh Meter | 1. Calibrate beginning and end of each day, and as necessary during use.<br>2. Check batteries - make sure they are fully charged.<br>3. Replace electrodes as needed.<br>4. Replace buffer solution regularly | 1. pH buffers<br>2. Batteries<br>3. Spare electrodes<br>4. Eh iodine solution |
| Conductivity Meter | 1. Check batteries<br>2. Calibrate beginning and end of each day, and as necessary during use.<br>3. Check redline and replace batteries if does not calibrate.<br>4. Clean probe regularly. | 1. Batteries |
| Dissolved Oxygen Meter | 1. Check Batteries<br>2. Calibrate in the field before each measurement or group of closely spaced measurements. | 1. Batteries |
| Dissolved Oxygen Meter Probe | 1. Condition probe in a water sample for as long as possilbe before use.<br>2. Recalibrate probe when the membrane is replaced. | |
| HNu Model Photoionization Detector | 1. Check battery and recharge when low<br>2. Check UV lamp<br>3. Check fan<br>4. Calibrate beginning and end of each day, and as necessary during use.<br>5. HNu's PID should be decontaminated or wiped down daily or after each use, as appropriate<br>6. Clean UV lamp and ionization chamber regularly | 1. Battery charger<br>2. Spare lamps |

**TABLE 2-5**

**PREVENTIVE MAINTENANCE FOR ANALYTICAL INSTRUMENTS**

| Instrument | Activity | Frequency |
|---|---|---|
| GC/MS | Change septum onGC | Daily |
| | Bake trap | Daily |
| | Clean source | Tune failure - record |
| | Change pump oil | Quarterly - record |
| | Clean injector/replace liner | SPCC failure - record |
| ICP | Torch inspection | Each use |
| | Clean torch and nebulizer | As needed - record |
| | Inspect filters | Daily |
| | Change filters | As needed - record |
| | Inspect pump tubing | Daily |
| | Change pump tubing | As needed - record |
| Leeman Mercury Analyzer | Change drying tubes | Daily |
| | Run aperture test | Daily |
| | Inspect tubes and reagents | Daily |
| Temperature Devices Refrigerator Drying ovens | Temperature | Daily or when used (Refrigerator 2X/day) |
| Weighing Balances | Clean pan | Each use |
| | Clean calibration | Daily - record |

**Table 3-1**
**Summary Table of Sampling Analysis Program**

| LOCATION | SOIL/SEDIMENT | | | SURFACE WATER/GROUNDWATER | | |
|---|---|---|---|---|---|---|
| | 8 RCRA METALS | VOLATILE ORGANIC COMPOUNDS (VOCs) | SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs) | 8 RCRA METALS | VOLATILE ORGANIC COMPOUNDS (VOCs) | SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs) |
| *Date Prepared: 4/12/02* | | | | | | |
| Section 1 | 2 | 7 | 7 | 4 | 4 | 4 |
| Section 2 | 2 | 8 | 8 | 4 | 4 | 4 |
| Section 3 | 2 | 8 | 8 | 4 | 4 | 4 |
| Section 4 | 2 | 7 | 7 | 2 | 2 | 2 |
| J-Pit Section 5 | 8 | 16 | 16 | 8 | 8 | 8 |
| Discretionary | 2 | 2 | 2 | 4 | 4 | 4 |
| QA/QC | 5 | 9 | 9 | 5 | 5 | 5 |
| Total | 23 | 57 | 57 | 31 | 31 | 31 |

**NOTE:**

Volatile Organic Compounds (VOCs) analyzed via USEPA SW-846 Method 8260B.
Semi-Volatile Organic Compounds (SVOCs) analyzed via USEPA SW-846 Method 8270A.
Metals (As, Ba, Cd, Cr, Pb, Se, and Ag) are analyzed via USEPA SW-846 Method 6010.
Hg is analyzed via USEPA Method SW-846 method 7471a for solid and 7470a for aqueous.

## Table 3-2. Sample Container, Preservation, Holding, and Volume Requirements

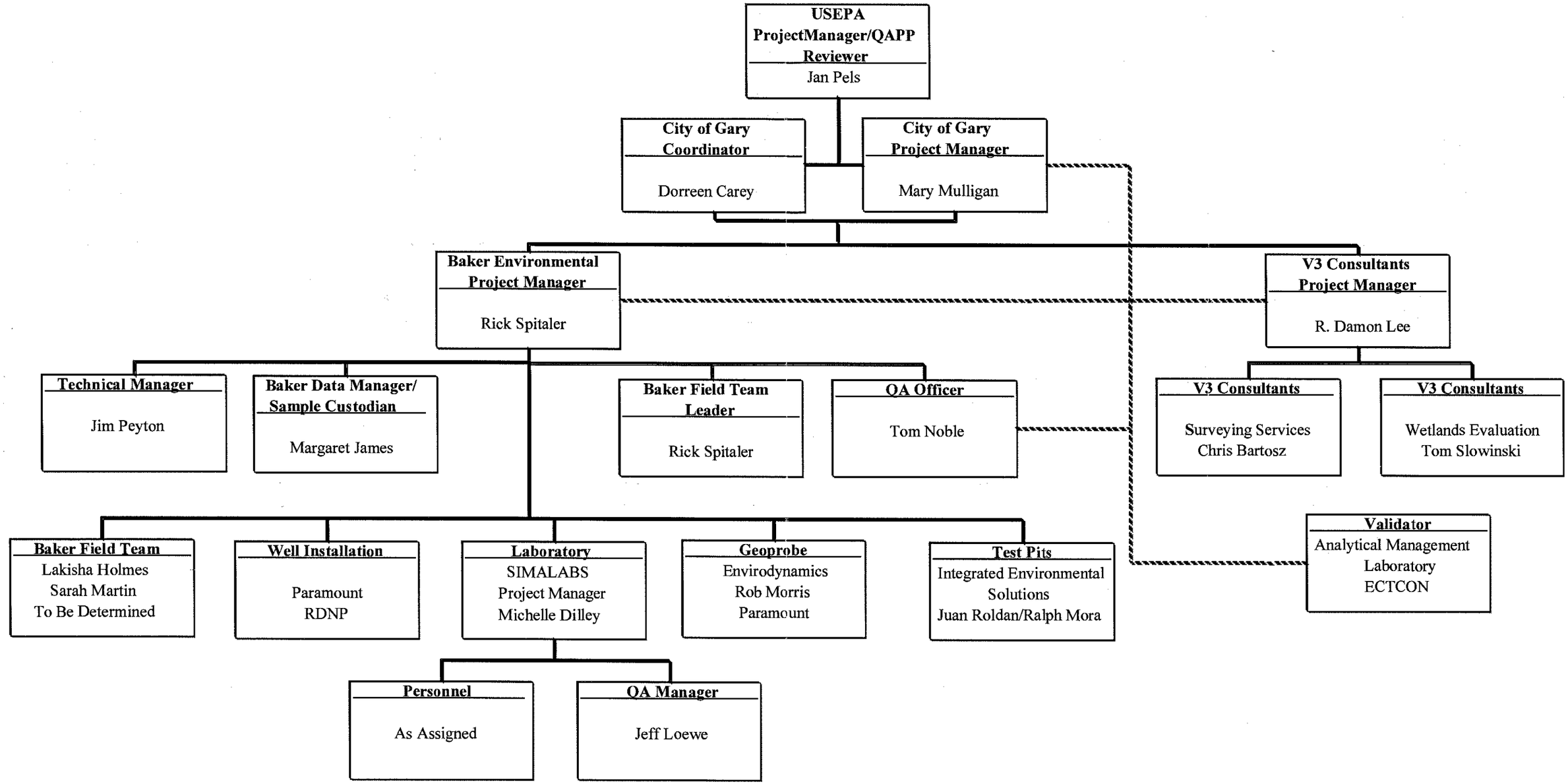
| Matrix | Name | Containers | Preservation | Holding Times | Minimum Sample Volume or Weight Soil/Water |
|---|---|---|---|---|---|
| *Date Prepared: 4/9/02* | | | | | |
| **Soil** | Metals, *except* Cr(VI) | 8 oz. WMJ (s) | Cool, 4 ± 2 °C | Metals (except Hg): 6 months<br>Hg: 28 days | 1 g/100 mL Hg 5 g/100 mL ICP/GF AA |
| | Semi-Volatile Organics | 8 oz. WMJ (s) | Cool, 4 ± 2 °C | 14 days for (s), 40 days ~~from collection~~ after extraction for aqu. | 30 g/ 1000 mL |
| | VOCs | 4 oz. WMJ (s) | Cool, 4 ± 2 °C | 14 days from collection | 5 g/25 mL, No headspace: |
| **Water** | Metals, *except* Cr(VI) | 1 L HDPE (aq) | 5ml of 35% $HNO_3$ to pH<2(aq):<br>Cool, 4 ± 2 °C | Metals (except Hg): 6 months<br>Hg: 28 days | 1 g/100 mL Hg 5 g/100 mL ICP/GF AA |
| | Semi-Volatile Organics | 2-1 L AG with Teflon-lined cap (aq) | Cool, 4 ± 2 °C | 7 days until extraction (aq)<br>40 days to analysis of extract | 30 g/ 1000 mL |
| | VOCs | 3-40 mL Borosilicate glass vial (aq) | 0.5 mL 36% HCl to pH<2 (aq): Cool, 4 ± 2 °C. For chlorinated waters, add 25 mg ascorbic acid per vial prior to sampling. | 14 days for (aq) | 5 g/25 mL, No headspace: |

Notes:

HDPE = High Density Polyethylene bottle
aq = Aqueous Sample
AG = Amber, Boston round, Glass Bottle
s = Soil Sample
WMJ = Wide Mouth Paragon Flint Glass Jar
Metals include: Ar, Ba, Cd, Cr, Pb, Hg, Se and Ag

# FIGURES

**FIGURE 1**
**PROJECT ORGANIZATION CHART**



# FIGURE 2 -1

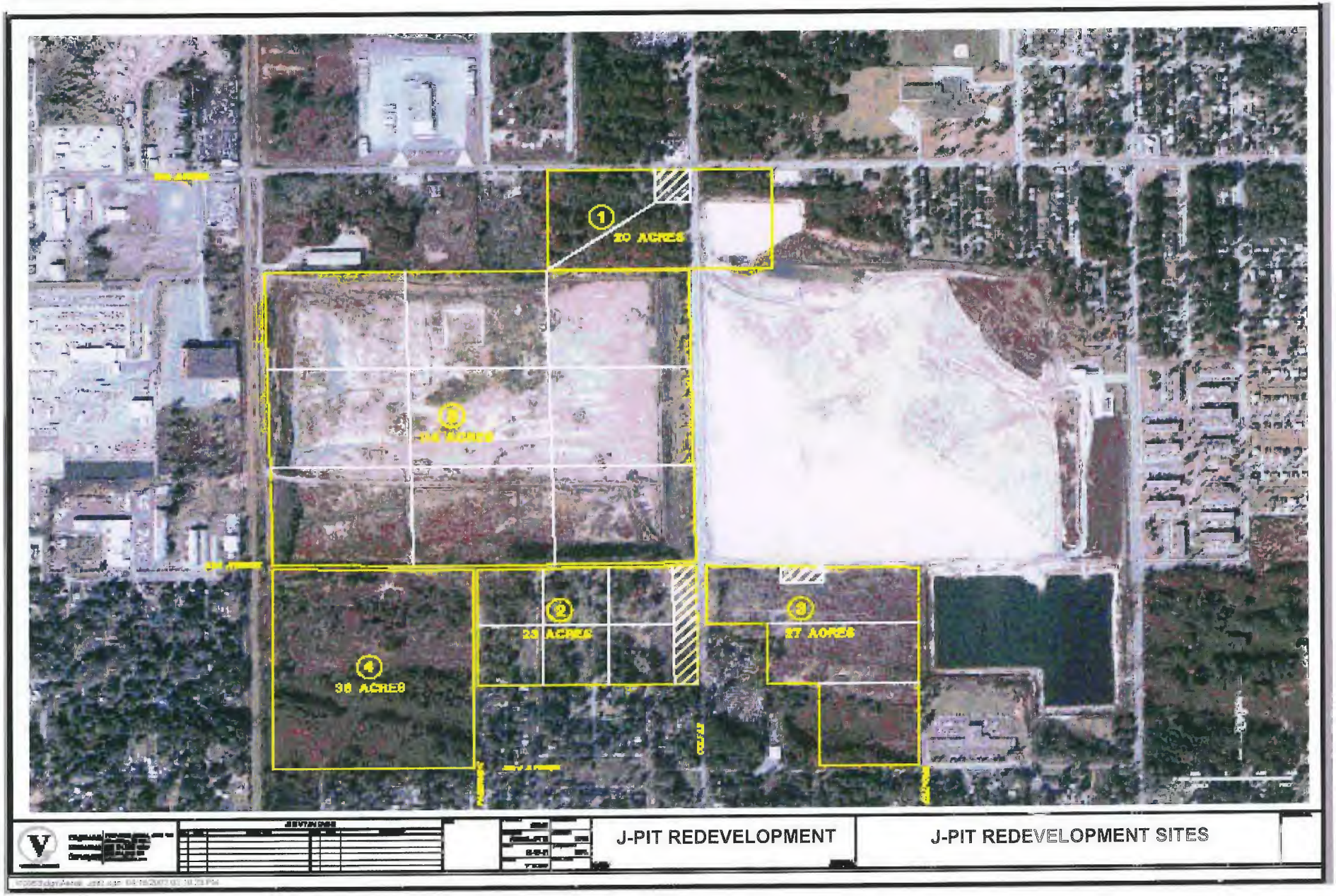

1
20 ACRES
2
25 ACRES
3
27 ACRES
4
30 ACRES
J-PIT REDEVELOPMENT
J-PIT REDEVELOPMENT SITES

**FIGURE 3**
**THE DATA QUALITY OBJECTIVES PROCESS**

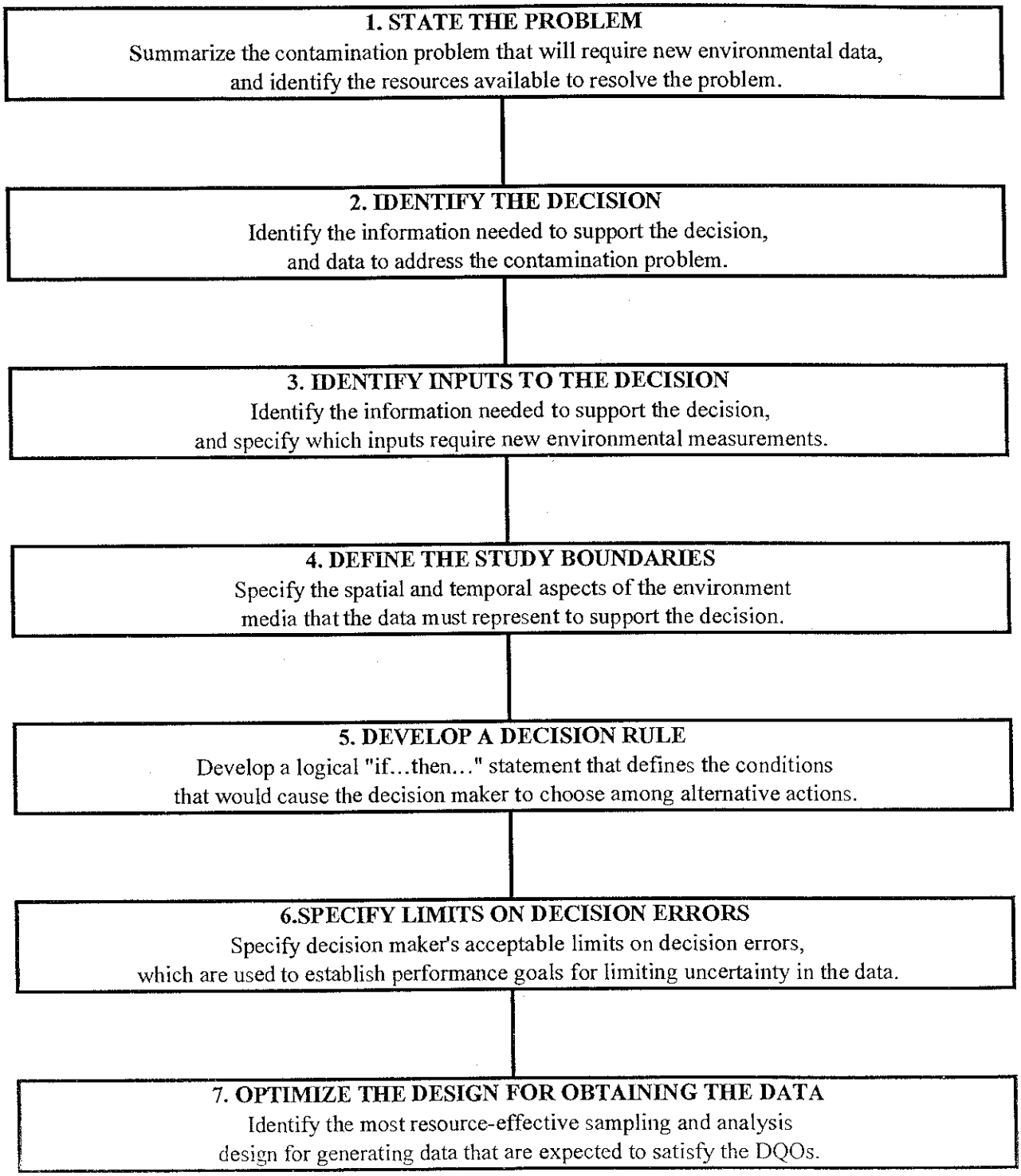
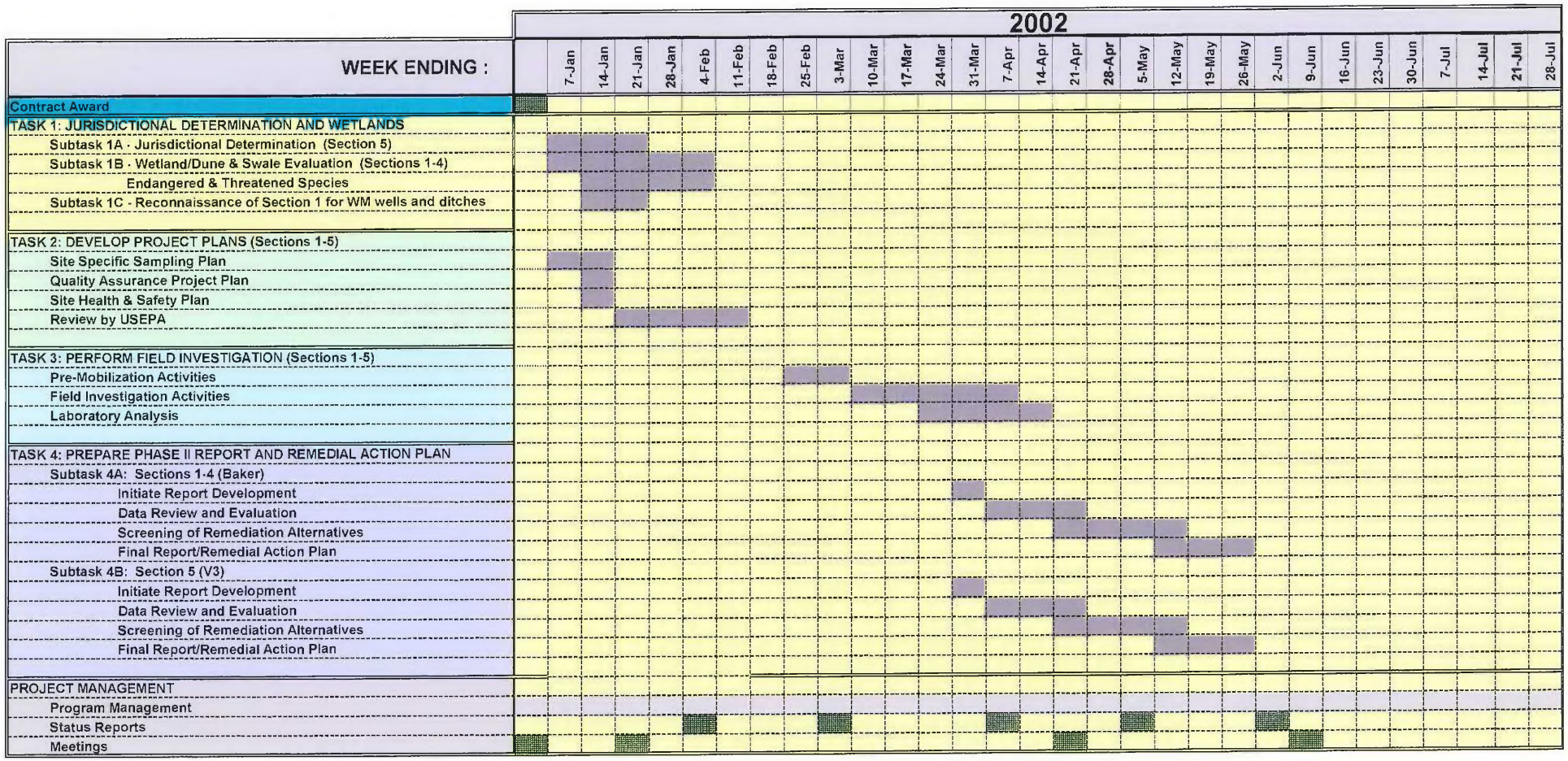


**Figure 4**

**Project Schedule**



**J-PIT REDEVELOPMENT SITE**
**PHASE II SITE INVESTIGATION**
**SAMPLING PROCEDURES**

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**J-PIT REDEVELOPMENT SITE**
**PHASE II SITE INVESTIGATION**
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# 1.0 INTRODUCTION

This document presents the Sampling and Analysis Plan (SAP) portion of the Quality Assurance Project Plan (QAPP) developed for the Limited Phase II Environmental Site Assessment (Phase II ESA) of the J-Pit Redevelopment Site (Site) located in the City of Gary, Lake County, Indiana (Figure 1-1). Baker Environmental, Inc. (Baker) has been selected by the City of Gary to perform the site investigation portion of the Phase II ESA.

This plan has been prepared to document the scope and objectives of the Phase II ESA investigation activities for the approximately 200-acre J-Pit Redevelopment Site and to ensure that Baker staff and subcontractors employ procedures/protocols in accordance with the July 1996 Indiana Voluntary Remediation Program (VRP) Resource Guide and industry standard engineering and geological practices. The SAP serves as a tool for assigning responsibilities and establishing appropriate sampling methods, rationale, and number of samples to be collected during the field investigation. This SAP is a guide to the project team as to what samples to collect, how to collect them, and what analytical methods to use for analysis.

## 1.1 Purpose and Objective

The overall objective of the Phase II ESA is to evaluate the presence of chemicals associated with recognized environmental conditions identified in the previous Phase I ESAs (or those determined during on-going field activities) that exceed IDEM VRP Tier II Residential Cleanup Goals for the purposes of developing the site as part of the Airport Redevelopment Zone. The SAP has been developed following the United States Environmental Protection Agency (USEPA) 7-step data quality objective process outlined in Section 1 of the QAPP.

Specifically, the SAP has been developed to provide guidance for all field activities including detailed descriptions of sampling and data collection methods to be used during various field tasks. The development of the SAP helps ensure that sampling and data collection activities are carried out in accordance with generally acceptable practices and to demonstrate that data obtained during the field investigation are of sufficient quantity and quality to fulfill project data quality objectives (See QAPP Section 1.0). The QAPP, SAP, and HASP have been developed to fulfill these objectives.

## 1.2 Sampling and Analysis Plan Contents

The SAP has been organized to present a comprehensive document, which describes the technical approach and field operations. The organization of the SAP includes the following Sections:

**Section 1.0**- Identifies the objective and the contents of the plan.
**Section 2.0** -Provides background information on the areas to be investigated.
**Section 3.0**- Details the technical approach and sampling methods.
**Section 4.0**- References

Appendix C of the QAPP provides a description of the standard operating procedures (SOPs) for each field investigation method. The SOPs are for reference purposes and are representative of Baker's standard operating procedures. For this reason, the duplication of site specific data is not included in SOPs. Procedural changes made to the SOPs in the field will be noted in logbooks and included in the final report. The text of the QAPP and the SAP were generated specifically for the Site; thus, if any inconsistencies arise between information provided in the QAPP or SAP as compared with any portion of the SOPs, the QAPP and SAP take precedence. It is understood that the SOPs may be altered, if deemed appropriate, due to unknown site conditions. Any variance from the SAP and SOPs due to field conditions will be noted in the field logbook and reported in the final report. Although not all of the SOPs are specifically mentioned in the work tasks, they will be implemented as needed.

## 2.0 SITE BACKGROUND

The following subsections provide a description of the site regarding location, history, and previous investigations.

### 2.1 Site Location

The area is comprised of the approximately 100 acre Greenspace Site (a former sand mine known as the J-Pit), and about 100 adjacent acres of abandoned and undeveloped property (Pilot Site). The Pilot Site property is comprised of four individual Sections located east of Burr Street, south of 15th Avenue, east of the E.J.& E. Railroad and north of 23rd Avenue. The Site is within Sections 11, 12, 13 and 14, Township 36 North, Range 9 West of the Third Principal Meridian. The Site is in an area of mixed usage including residential subdivisions, undeveloped properties, and some industrial areas (Figure 2-1).

### 2.2 Site History

In May 2000, the City of Gary received a USEPA Brownfields Assessment Demonstration Pilot Grant. to identify, assess, and begin the redevelopment process for brownfields located within the 200-acre J-Pit Redevelopment Site, which is a portion of the 8,200-acre Airport Development Zone. The Site is at the center of a proposed light industrial, commercial and greenspace complex. One of the proposed restoration plans for the J-Pit is to fill in the former excavation with groundwater to form a lake. The banks of the new lake would be restored through plantings, construction of wetlands, and a trail would be constructed around the lake to connect to the industrial/commercial complex and a new city park to be located adjacent to the site.

In July 2001, Phase I ESAs were performed by Environmental Design International, Inc for the approximate 100-acre J-Pit Greenspace Site and adjacent Pilot Site parcels. The Pilot Site is comprised of four Sections as described below:

- Section 1: This Section consists of approximately 20 acres and is bound on the west by Hobart Street, on the north by 15th Avenue, on the east by Dallas Street, on the southeast by the closed Gary Municipal Landfill and on the southwest by the J-Pit.

- Section 2: This Section consists of approximately 15 acres south of the J-Pit and is bound on the south by 21st Avenue, on the west by Colfax Street, on the north by 22nd Avenue, and on the east by Fairbanks Street. The eastern 1/5$^{th}$ of the Section contains a parcel formerly occupied by an auto scrap yard.

- Section 3: This Section lies directly south of the closed Gary Landfill and consists of approximately 30 acres bound to the east by Colfax Street, Hamlin Street and King Street; to the south by 21st Avenue; to the north by 23rd Avenue; and to the west by Calhoun Street.

- Section 4: This Section encompasses roughly 40 acres and is bound to the north by 21st Avenue, to the south by 23rd Avenue, to the west by Fairbanks Street, and to the east by the EJ&E Railroad line. Only the northern 20 acres of this Section are included in the Phase II ESA. The remaining 20 acres are a wetland area that is not included in the redevelopment project.

Individual Phase I ESA reports were prepared for each of these Sections and the Greenspace Site (i.e. five total reports). Several Recognized Environmental Concerns (RECs) were documented in the Phase I ESA Reports. In general, some level of Phase II sampling was recommended for every parcel due to their proximity to the closed Gary Municipal Landfill, two Superfund sites (Midco I and 9th Avenue Dump), several Leaking Underground Storage Tank (LUST) sites, and specific areas judged to constitute RECs as outlined below.

Section 1: Although the parcel is predominantly vacant, two small buildings were observed on the west side (south of 15th Avenue), an area historically used as a junk yard. Surface and buried refuse, tires, an old bus, metal, plastic, and empty 55-gallon drums were observed.

Section 2: Structures and scattered piles of debris including shredded tires, two empty 55-gallon drums, and scrap metal were observed on the northeast side in the area of the former Paul's Auto Yard (a scrap yard once located at 2124 Colfax); and LeRoy's Used Cars (previously located at 2150 Colfax). The structures at Paul's Auto Yard have been demolished and the ground leveled. As a consequence buried material exist at the Site. In addition, scattered rubbish was observed along the boundary line with the J-Pit area to the north.

Section 3: The north and northwest portions of this parcel reportedly were used in the past for car repairs and dumping. Prior to 1990, the Gary Municipal Landfill did not have a slurry wall along the approximately 1,200 foot common property line. Active junkyards are located adjacent to southwest. Bivona, Inc., to the southeast was listed on the Toxic Release Inventory (TRI) for Trichloroethane (TCE), and currently is enrolled in the IDEM VRP for chlorinated solvents. Debris, scattered piles of rubbish including shredded tires, and stressed vegetation were observed at various locations throughout the site.

Section 4: Partially buried construction debris was observed near the boundary fence of the J-Pit (north border); and railroad tracks are located along the west border.

Greenspace Site (J-Pit): At one time this Section was operated by Waste Management as a waste area. An oily, rust-colored unknown substance was observed in an approximately 1,300 foot excavated ditch located on the north side of the pit. Debris was observed along the perimeter including tires, concrete piping, concrete, scrap metal, plastics, and some buried debris. Three monitoring wells were observed partially submerged in water on the east side, as well as one well in the southeast corner and one in the northeast corner of the site. A groundwater and leachate collection system for the closed Gary Municipal Landfill is located on the east side. The system is seven feet in diameter, with three inlets from the south, north and northeast. Inlet piping runs under vegetation in a ditch extending along the east end of the parcel. The west wall of this ditch is used as a service road. Much of the J-Pit is flooded during the winter and spring months Stressed vegetation was observed along the wall of the ditch, five feet from the bottom during the Phase I ESA.

## 2.3 Aerial Photographic Review

As part of the evaluation of site background, Baker reviewed aerial photographs from 1938, 1958, 1965, 1973, 1987 and 1992, acquired from the records of the Soil Conservation District for Lake County, Indiana. In 1938 the site was part of a largely undisturbed dune and swale topography typical of the majority of the region. By 1958 the J-Pit sand mine (Section 5) and the Gary Municipal Landfill were visible and apparently active. Subsequent photographs indicate periodic flooding of the J-Pit and the emerging industrial and semi-industrial development of the surrounding

vicinity. The surrounding properties (Sections 1 to 4) remained largely undeveloped, except for the construction of subdivisions in Section 2 and some businesses along Colfax Avenue.

## 2.4 Site Geology/Hydrogeology

According to the 1992 United States Department of Agriculture (USDA) Soil Conservation Service (SCS) Soil Survey of Lake County, Indiana, the site and immediate area are underlain by the Oakville-Tawas complex and the Tawas Muck soils. The Oakville-Tawas complex is comprised of about 45 percent Oakville sand and 45 percent Tawas Muck. The Oakville sand is a black to yellowish brown, excessively drained, fine sand located on narrow ridges. The Tawas muck is a black, deep, very poorly drained organic muck underlain with a grayish-brown loose sand in depressions between the Oakville sand ridges (1992, USDA, Lake County Soil Survey). The surface at the site has been heavily altered by sand mining, filling, grading, spreading gravel and stockpiling activities.

The site is located at the boundary of lacustrine deposits of the Calumet Lacustrine Plain physiographic region and the Toleston Beach complex. The Calumet Lacustrine Plain has been heavily altered due to industrial and residential development. Where undisturbed, the area is dominated by three relict dune-capped beach ridges separated by broad inter-ridge marches. The Toleston Beach complex extends from between the Little Calumet River and the Grand Calumet River to Lake Michigan and is characterized by linear ridges of unified parabolic dunes interspaced with interdunal swamps (dune and swale). The unconsolidated materials were deposited in the Wisconsin age, with a combined action of ice, wind and water. The glacial deposits in the vicinity and surrounding area have an estimated thickness of 150 feet (1994, Department of Environmental Resources Water Resource Availability in the Lake Michigan Region, Indiana).

The site is within the Calumet Aquifer System consisting of fine to medium sand with beach gravel beds. Discontinuous beds of silt, clay and peat deposits confine the aquifer in some locales. The aquifer is considered highly susceptible to surface contamination due to the lack of a clay cap or separator beds (1994, Department of Environmental Resources Water Resource Availability in the Lake Michigan Region, Indiana). Regional groundwater flow is anticipated to be to the north toward the Grand Calumet River and Lake Michigan, but due to the location of the site between the Grand Calumet and Little Calumet Rivers, local groundwater flow may differ.

The unconsolidated soils are underlain by the Wabash Formation within the Kankakee Arch. The Wabash Formation is comprised of reef and inter-reef Silurian dolomite, dolomitic limestone and argillaceous limestone (1994, USGS Hydrogeologic Atlas of Aquifers in Indiana).

## 2.5 Future Land Use

The future land use is to redevelop the 200-acre J-Pit Redevelopment Site, within the 8,200 acre Airport Development Zone. Included in development process for the J-Pit and Pilot Site is to assess and maintain area wetlands, and to fill the J-Pit with groundwater to form a lake. The banks of the new lake are to be maintained through the planting and construction of wetland habitat. A trail would be constructed connecting the industrial/commercial complex and the new city park adjacent to the site. The development process will create jobs, provide training opportunities for local residents, and establish safe and productive industries. The redevelopment process will also facilitate the infusion of tax revenue to the city, further improving the quality of life for residents.

## 3.0 TECHNICAL APPROACH AND GENERAL FIELD OPERATIONS

This section presents an overview of the field investigation including specific details of the investigations. The field investigation shall reflect the criteria to meet the Phase II ESA objectives identified in Section 1.0 and the QAPP.

### 3.1 Mobilization

The field investigation will be initiated through mobilization activities including equipment procurement, utility clearances, location of decontamination and drum storage areas, etc. All subsurface exploration, field equipment, and decontamination equipment and associated materials will be mobilized by Baker's subcontractors. Sample labels, containers, identification numbers, and tracking documents will be provided by the contracted laboratory. Remaining sampling equipment and other supplies will be collected and provided by the Baker field team.

### 3.2 Field Personnel and Responsibility

Baker intends to staff the investigation with a Project Manager, a Project Geologist serving as the Field Team Supervisor, and a Field Sampling Team. Additionally, administrative and technical support will be available for various tasks of the project. Figure 3-1 of the QAPP presents the Project Organization Summary. A brief summary of staff employed for the implementation of the SAP is provided below. For a further description of key personnel for the project, please refer to the QAPP and HASP (Appendix A of the QAPP).

The Project Manager will have as primary responsibilities: monitoring technical, cost and schedule performance; orchestrating Baker's overall Quality Assurance (QA) efforts, document reviews and cost/schedule reviews; and maintaining close communication with the City of Gary, USEPA and IDEM project representatives.

The Project Geologist will locate the sample and monitoring well locations and be responsible for on-site technical aspects of the project. He will be the primary contact with City of Gary Project Manager throughout the field activities. The Project Geologist will also maintain the field log book, maintain all sample documentation, take project photographs, and conduct and supervise sample

collection and sample packaging.

The Field Team Supervisor will be responsible for directing all investigation activities and accomplishing the work in accordance with the SAP. The Project Geologist for this project will also serve as the Field Team Supervisor.

The Health and Safety Coordinator will be responsible for assuring compliance with the HASP. In addition, he will use air-monitoring instruments to indicate whether or not an upgrade in the level of personal protection is necessary. The Project Geologist for this project will also serve as the Health and Safety Coordinator. The HASP is bound as a separate project document (Appendix A).

The Sampling Team will assist the Project Geologist as needed with sample collection, packaging, and calibration of field instruments.

## 3.3 Field Activities

Activities associated with the field investigation include:

- GeoProbe® and Soil Sampling Investigation
- Test Pit Soil Sampling
- Sediment and Surface Water Sampling
- Installation of Groundwater Monitoring Wells
- Groundwater Sampling

Each of the above field activities are discussed in detail in the following sections for each of the five Sections of the Site. Optional field activity is presented for use during potential subsequent sampling events.

### 3.3.1 GeoProbe® Sampling

The first phase of sampling will be accomplished through the use of a GeoProbe® rig collecting surface and subsurface soil samples. The objective of the GeoProbe® sampling is to determine whether a release associated with the previously discussed areas of environmental concern exceed

the IDEM VRP Tier II cleanup goals. For each area, a base number of GeoProbe® borings have been determined for the initial sampling. General sampling locations for each area are shown on Figure 3-1. Actual sample locations will be determined in the field based upon observed areas of elevated environmental concern (i.e., buried drums, debris piles, etc.). In the event that additional areas are encountered during the field activities, GeoProbe® borings may be relocated, or additional borings added to the investigation, pending authorization from the City of Gary Project Manager.

GeoProbe® borings will be located at each of the five Sections (Figure 3-1) as follows:

- Section 1: a total of seven GeoProbe® borings will be advanced. The focus will be on the area west of Colfax Street.

- Section 2: a total of five GeoProbe® borings will be advanced focused on addressing local community concerns along the southern border.

- Section 3: a total of eight GeoProbe® borings will be advanced. The focus will be on the eastern border with the Bivona VRP site and northern border with the Gary Municipal Landfill

- Section 4: a total of four GeoProbe® borings will be advanced. The focus will be on the area west of Colfax Street.

- Section 5: a total of six GeoProbe® borings will be advanced. The focus will be on the western and the southern borders where the native sands still exist above the upper clay formation.

At each sampling location, the GeoProbe® sampler will be fitted with a new acetate sleeve and advanced at 4-foot intervals to groundwater (estimated at a depth of 5 to 15 feet bgs). Each 4-foot GeoProbe® soil sample will be opened at the surface, screened using a photoionization detector (PID) and logged by the on-site geologist. One soil sample will be selected from each borehole based upon PID readings, and visual and field indications of the highest soil contamination in each boring. In the event that no indications are observed, Non-VOC soil samples will be collected based upon on the most likely location of COC based upon the soil type and COC characteristics or other

visible signs of contamination, per the observations of an Indiana licensed, professional geologist.

Soil will be transferred from the GeoProbe® acetate sampler into laboratory supplied jars with a decontaminated stainless steel spoon. Soil samples will be placed on ice in a cooler pending submittal to the laboratory. The GeoProbe® sampling procedures are presented in Appendix B of the QAPP. Following the sampling activities, each GeoProbe® borehole will be backfilled to the ground surface with bentonite.

### 3.3.2 Test Pit Trenches

The test pits are to be conducted following the GeoProbe® phase of the investigation. Test pit trenches will be conducted in the former Paul's Auto Yard to evaluate subsurface conditions of reported historical buried materials or at any other location identified during the GeoProbe® investigation. Test pits and trenches permit detailed exploration of the nature and extent of contamination of in-situ materials, and the characteristics and stratification of near surface materials using standard equipment to a common depth of up to 15 feet. Test pits normally have a width ranging from two to ten feet or greater, depending on the objectives of the excavation and the equipment used. While test trenches are elongated test pits, usually three to six -feet wide and extending for any desired length.

Excavation shall commence by removing lifts of no more than 6 to 12 inches of soil. The test pits and trenches shall be logged and sampled by the Project Geologist. The field logs shall provide the exact dimensions and location of the test pits or trenches as well as soils classification and description in accordance with the Baker standard operating procedures in SOP F001 (Appendix C). Samples shall only be collected from material in the equipment backhoe bucket, or from the excavated material. Prior to and after each test pit excavation or sampling location, equipment shall be thoroughly decontaminated. After inspection and completion of the appropriate test pit logs, backfill material will be retumed to the pit under the direction of the Project Geologist. Backfilling of trenches and test pits is normally accepted practice to reduce immediate site hazards and minimize the potential for rainwater accumulation and subsequent contaminants migration.

During the test pit excavations, three soil samples will be selected for analysis based upon PID readings, and visual and field indications of the highest soil contamination.

### 3.3.3 Surface Water Sampling

Surface water sampling will be collected within the J-Pit at the approximate locations identified on Figure 3-1. In addition to the surface water samples collected in the J-Pit, two surface water samples will be collected from ditches immediately to the south of Section 2. The locations will address local residents concerns and will be determined by the City of Gary. Sediment/surface soil samples may replace surface water samples within the pit if no surface water is present at the time of sample collection. Water samples will be collected from the middle of the water column. The sample will be collected at least six inches above the bottom to avoid stirring up the sediment and biasing the sample. Care will be taken to minimize sediment disturbance while collecting surface water samples.

Samples will be collected using one of the following sampling devices: dipper cup, sampling bomb, Kemmerer sampler, or directly into laboratory bottles, with preservation at the surface, based upon site conditions at the time of the sampling event.

The Kemmerer sampler (preferred method) is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while being lowered in a vertical position to allow free passage of water through the cylinder. A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles.

Samples may be collected by immersing either the approved sample container or a glass or nalgene beaker into the water. Sample bottles (or beakers) which do not contain preservatives will be rinsed at least once with the water to be sampled prior to sample collection. Care will be taken to avoid excessive agitation of the water, which may result in the loss of volatile constituents. Additionally, samples for VOC and SVOC analyses will be collected first, followed by the samples for RCRA metals. Measurements for temperature, pH, specific conductance, or other field parameters, as appropriate, may be collected immediately following sample collection for laboratory analyses.

Samples slated for VOC, SVOC and RCRA metals analysis will be collected directly into laboratory supplied bottles and placed on ice pending shipment to the laboratory. All samples will be collected

according to SOP 105 and handled as described in SOP F301 (Appendix C of the QAPP). The sampling locations shall be marked via wooden stakes placed at the nearest bank or shore.

### 3.3.4 Sediment Sampling

Sediment sampling will be collected within the J-Pit at the approximate locations identified on Figure 3-1. In addition to the sediment samples collected in the J-Pit, two sediment samples will be collected from ditches immediately to the south of Section 2.

Following collection of surface water samples, sediment samples may be collected from the top of the sediment to a depth of 0.5 feet. As with surface water samples, sediment samples will be obtained using on-shore techniques. Sampling personnel shall use judgment in removing large plant fragments to limit bias caused by bioorganic accumulation.

Sediment sampling will be conducted using one of the following sampling devices: hand auger, sediment core samples, Eckman or Ponar dredge, based upon site conditions at the time of the sampling event. Sediment will be placed in a stainless steel bowl and allowed to drain by partially decanting accumulated liquid.

Sediment corer or bucket (hand) auguring is a viable method for collecting sediment samples in shallower locations. The auger hole is advanced one bucket or core at a time, to a depth of 0.5 feet. The same bucket auger or sediment corer is used to advance the hole, as well as collect subsequent samples in the same hole.

Dredges are generally used to sample sediments which cannot easily be obtained using coring devices (i.e., coarse grained or partially cemented materials), or when large quantities of materials are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a messenger.

The Eckman dredge performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. The Ponar dredge is one of the most effective samplers for general use on all types of substrates. Access to the secured sample through the covering screen permits

sampling of the secured material with coring tubes or Teflon scoops, thus minimizing the chance of metal contamination from the frame of the device.

Samples will be transferred into laboratory supplied bottles, placed on ice and delivered to the contract laboratory for analysis. All samples will be collected according SOP 105 and handled as described in SOP F301 (Appendix C of the QAPP). Following sampling, staking of location and logging of sampling information shall be conducted.

### 3.3.5 Monitoring Well Installation

New monitoring wells will be installed, following the previous field activities using either hollow-stem augers or direct push techniques depending on the projected depth of individual wells. Wells to be installed at depths of 20 feet bgs or more will require hollow-stem augers to advance the borehole.

Split-spoon samples will be collected to the boring terminus based upon reviewing boring logs in general accordance with ASTM Method D 1586-84. Alternatively, where groundwater is encountered less than 20 feet bgs, direct push wells may be installed. The wells will be installed directly into the formation with a one-inch sand filter pack. The direct push, or natural filter, wells may have higher percentage fines and lower relative hydraulic conductivities, as compared to standard two-inch well installation. The wells will be developed as described below, but may require additional well volumes. Monitoring wells will be installed in each of the five Sections as follows (Figure 3-2).

- Section 1: Four monitoring wells will be advanced with focus on the area west of Colfax Street.
- Section 2: Four monitoring wells will be installed, with one in the area of the former scrap yard, two along the residential properties at the south of the Section, and one along the north side in the anticipated down gradient direction of the scrap yard.
- Section 3: Four monitoring wells will be installed, one along the eastern border with the Bivona VRP site, one on the northern border with the landfill, one on the eastern border and one on the southern border.
- Section 4: Two monitoring wells will be installed: one in the central area of the Section and one across from the railroad tracks.
- Section 5: two monitoring wells will be installed. These will be installed along the western and northwest borders. Since the J-Pit is at the bottom of the upper overlying sand aquifer,

only a limited number of well locations are feasible in the J-Pit. As a consequence staff gauges may be used to assist in developing hydrogeologic data for groundwater flow diagrams.

- In addition two existing shallow wells at the City of Gary Landfill property will be evaluated and, if feasible, selected for analysis.

Installation of the monitoring wells will consist of 2-inch diameter, Schedule 40 PVC screen and riser. The screen will be 10 feet long with 0.010-inch machined slots. The annulus surrounding the well screen will be backfilled from the bottom of the borehole to a minimum of two feet above the top of the screen with 10 to 12 mesh silica sand. A two-foot thick bentonite seal will be placed above the sand backfill. The remaining annulus surrounding the well riser pipe will be tremie grouted with a cement/bentonite grout or suitable grout, as determined by the on-site geologist. Selected locations may be completed as flush-mounted wells or with stick-up protective casings depending upon their location relative to roadways and property constraints. Both completion techniques will require that the final casing (flush or stick-up) will be embedded in a concrete pad and supplied with a locking cap and lock. The pads will be sloped so as to provide positive drainage away from the well.

A description of standard operating procedures for drilling and installing monitoring wells (F100 through F103) are presented in Appendix C of the QAPP.

The newly installed monitoring wells will be developed no sooner than 24 hours following installation. Prior to development, measurements of the static water level and total depth will be recorded for well volume calculations. A minimum of three and a maximum of five well volumes will be evacuated either through bailing or pumping. The field parameters of pH, specific conductance and temperature will be recorded in the field books after each well volume has been evacuated. The wells will not be considered developed until stability of the field parameters (typically less than 10 percent change between three successive measurements) or evacuation of a maximum of five volumes has occurred. All discharge waters will be handled in accordance with SOP F504 - Handling of Site Investigation Derived Waste (Appendix C of the QAPP), as discussed in Section 3.3.6.

### 3.3.6 Groundwater Sampling

An initial groundwater sampling event will be conducted one week following the development of the new monitoring wells. Figure 3-2 shows the location of these wells. Prior to purging, static water level and total depth measurements will be recorded for well volume calculations. As with well development, a minimum of three and a maximum of five well volumes will be evacuated prior to sampling. The preferred well evacuation/sampling method is the use of a submersible pump. Other acceptable well evacuation methods include a peristaltic pump or bailer.
Measurements of pH, specific conductivity, and temperature will be recorded after the evacuation of each well volume. The wells will not be considered sufficiently purged until stability of the field parameters (typically less than 10 percent change between three successive measurements) or removal of a maximum of five volumes has occurred.

Each groundwater sample will be analyzed for the following parameters:

- Volatile organic compounds (VOCs) - SW-846 Method 8260
- Semi-volatile organic compounds (SVOCs) - SW 846 Method 8270
- RCRA metals - SW-846 Methods

RCRA metals will be collected in preserved sampling bottles. The SOP for groundwater (F104) sample collection is presented in Appendix C of the QAPP.

### 3.3.7 Site Survey

Following field activities, the sample locations will be measured for horizontal control by V-3 Consultants personnel. New monitoring wells will be surveyed to already existing monitoring wells for vertical and horizontal control. The survey will be performed with standard, acceptable surveying practices as required by the State of Indiana. All surveying will be conducted under the supervision of a Registered Land Surveyor licensed in the State of Indiana.

### 3.3.8 Decontamination and Waste Handling

Prior to field activities, between each boring location, and prior to leaving the site, hollow- augers,

mud rotary tooling, and all associated drilling and sampling equipment will be steam cleaned in order to prevent cross-contamination. During field activities, tooling, split-spoon samplers, and associated sampling equipment will be decontaminated via the following six step procedure:

- Alconox soap and potable water wash
- Potable water rinse
- Isopropyl alcohol or methanol wash
- Distilled water rinse
- Nitric acid (5%) wash
- Triple distilled water rinse

This same sequence for decontamination will be used for smaller equipment (such as spoons, trowels, and bailers), but without steam cleaning. Personal decontamination steps are described in the HASP (Appendix A of the QAPP).

Purge water and associated decontamination fluids, will be placed into properly labeled 55-gallon drums or polyvinyl tanks, and stored in a secured area within the property pending analytical results. Soil cuttings may be drummed or stockpiled on plastic sheeting, covered with plastic sheeting and disposed with the soils to be excavated. Health and safety disposables, such as sampling gloves, paper towels, or other materials which may come in contact with potentially contaminated materials will be placed in suitable containers and stored with the solid and fluid wastes pending analytical results.

Purged water and investigation derived waste (IDW) will be discharged into fifty-five gallon drums and stored on-site until the status of groundwater quality is determined, for later disposal by the City of Gary. Baker will collect representative samples during the generation of the IDW for the City of Gary.

Appendix C of the QAPP presents SOPs for Decontamination of Drilling Rig and Monitoring Well Chemical Sampling (F501), Field Analytical Equipment Decontamination (F502), and Handling of Site Investigation Wastes (F504), respectively.

## 3.4 Sample Analysis and Preservation

The samples will be analyzed for the following parameters (See Table 3-1 and Table 3-2):

- Volatile Organic Compounds (VOCs) analyzed via USEPA SW-846 Method 8260B.
- Semi-Volatile Organic Compounds (SVOCs) analyzed via USEPA SW-846 Method 8270A.
- Metals (As, Ba, Cd, Cr, Pb, Se, and Ag) are analyzed via USEPA SW-846 Method 6010Hg is analyzed via USEPA Method SW-846 method 7471a for solid and 7470a for aqueous.

All samples will be preserved with ice to a temperature of 4° Celsius prior to transportation to the analytical laboratory (routine groundwater samples). Aqueous samples (groundwater, Quality Assurance/Quality Control [QA/QC] samples and blanks) to be analyzed for VOCs will be preserved with hydrochloric acid to a pH of less than 2. Aqueous samples to be analyzed for RCRA metals will be preserved with nitric acid (HNO3) to a pH of less than 2 and field filtered prior to preserving the sample.

The number and type of samples for each sampling event are described on Table 3-1. Appendix D of the QAPP presents the Sample Preservation SOP.

## 3.5 Quality Assurance/Quality Control

During the field activities, an appropriate number of QA/QC samples will be collected in order to verify the accuracy of data supplied by the on-site laboratory. Duplicate samples will be collected at a ratio of one duplicate in 10 samples (10 percent) and one matrix spike/matrix spike duplicate (MS/MSD) in 20 samples (5 percent). This ratio of QA/QC sample collection will be maintained during each subsequent routine sampling event. In addition to the duplicates and MS/MSD samples, an appropriate number of rinsate blanks and trip blanks will be collected as specified in the QAPP.

## 3.6 Sample Numbering

Subsurface soil samples will be designated with a SB prefix (for Soil Boring)) and boring number followed by the individual sample number from that location. An example of this would be SB01-01: where SB indicates a Soil Boring; 01 indicates the first boring in the series; and -01 designates the first sample collected from that boring.

Monitoring wells installed during this investigation will have an MW prefix followed by the number designation. Groundwater samples collected from the initial sampling event, conducted after the installation of the new wells, will be designated simply by the well number (i.e., MW-01). The surface water samples will have an SW prefix, and sediment samples a SD prefix.

Samples from which QA/QC samples were collected will have the suffix of D for duplicate, and MS/MSD for matrix spike/matrix spike duplicate attached to the sample designation. Rinsate blanks will be designated by the prefix RB and a sequential number, and trip blanks will be designated by a TB prefix.

### 3.7 Chain-Of-Custody, Sample Packing and Shipping

Proper chain-of custody documentation will be maintained for all samples from the time of collection until they are shipped to the laboratory. Chain-of-custody sheets accompanying the samples will contain the following information: project number, sampler(s) name, sample numbers, number of containers, method(s) of preservation of samples, date and time of sample collection, analysis(es) requested, date and time of transportation to the laboratory, method of transportation, and any other information pertinent to the samples. Sample documentation will be prepared in accordance with Baker's SOP for sample Chain-of-Custody (F302), which is included in Appendix C of the QAPP.

### 3.8 Documentation

A single notebook will be dedicated to the investigation and will serve as a daily logbook detailing the weather and activities of the day, including work accomplished, those present on site, and technical issues such as sample numbers, descriptions of sample locations, any problems encountered during sample collection and preservation methods. The Field Logbook SOP (F303) is presented in Appendix C of the QAPP.

### 3.9 Field Change and Corrective Action

If changes become necessary due to field conditions (e.g., weather problems, obstruction to sampling locations), the proposed change will be communicated from Baker's Field Team Supervisor to

Baker's Project Manager, and then to the City of Gary Project Manager. Upon mutual agreement of the best method of solving the problem, the method will be implemented and the changes documented, with the documentation placed in the project file.

### 3.10 Field Instrument Calibration

Equipment calibration will be performed at the frequency and using the directions recommended by the manufacturer of each piece of equipment. Calibration will be performed daily, in the morning, prior to initiation of field activities. Refer to Appendix C of the QAPP for PID (F203) and O2/LEL Meter (F206) SOPs, respectively.

## 4.0 REFERENCES


U.S. Environmental Protection Agency (U.S. EPA). *Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan*. Revision 0 June, 2000.

U.S. Environmental Protection Agency (U.S. EPA). 1985. *NEIC Policies and Procedures.* EPA 0 33019-78-DDI-$. Revised June 1985.

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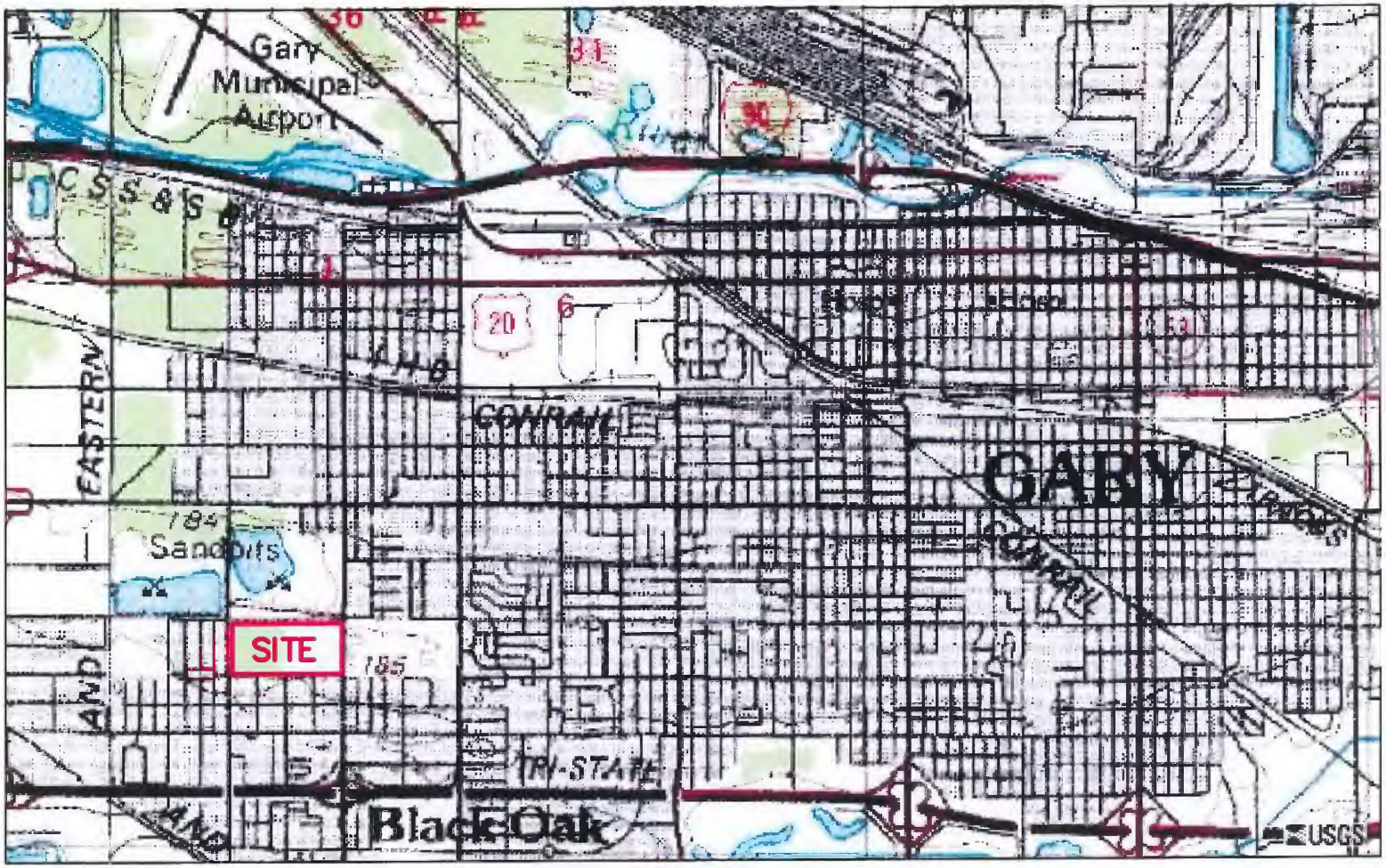
U.S. Environmental Protection Agency (U.S. EPA). *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans.* QAMS-005/80.

# TABLES

# FIGURES




CONTOUR INTERVAL 10 FEET

US GEOLOGICAL SURVEY, JULY 01, 1991

GARY/HIGHLAND QUADRANGLE

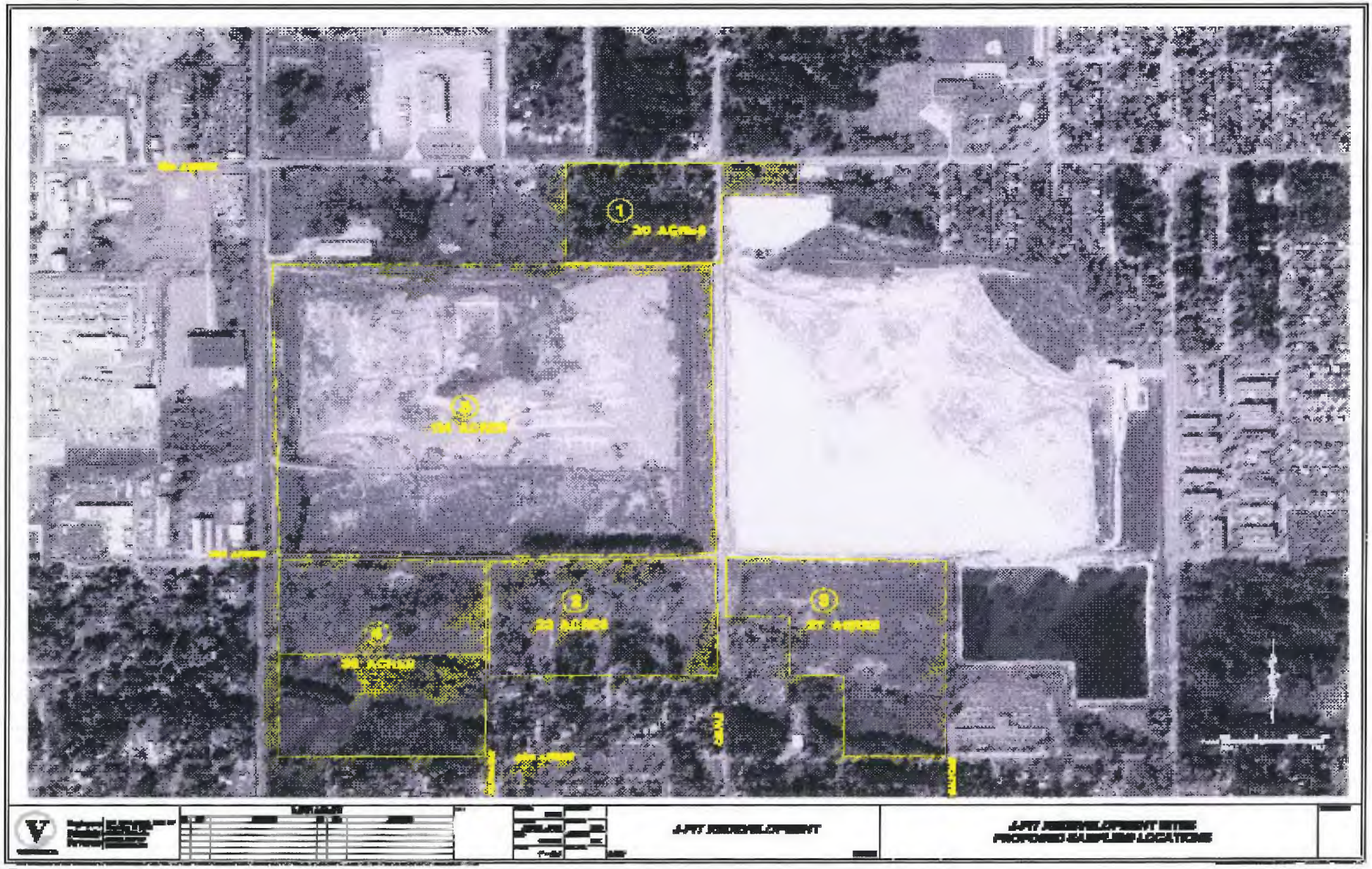
| | |
|---|---|
| APPROX. SCALE: | 1" = 45,000' |
| S.O. No. | 25290-000-0000 |
| DATE: | MARCH, 2002 |
| FILE: | 25290F01.DWG |

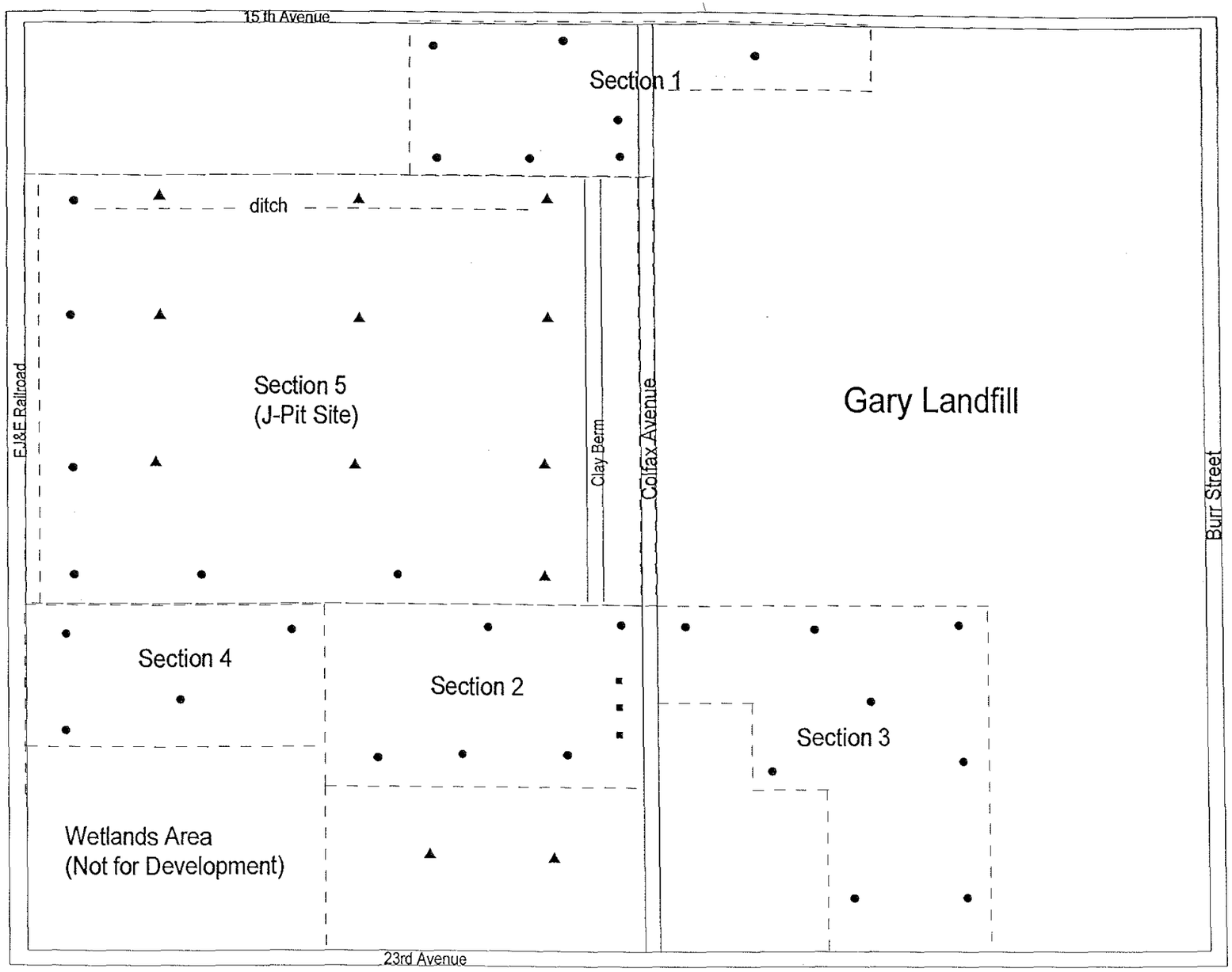
# FIGURE 1-1. SITE LOCATION MAP
# J-PIT REDEVELOPMENT SITE PHASE II ESA
# CITY OF GARY
# GARY, INDIANA

Baker

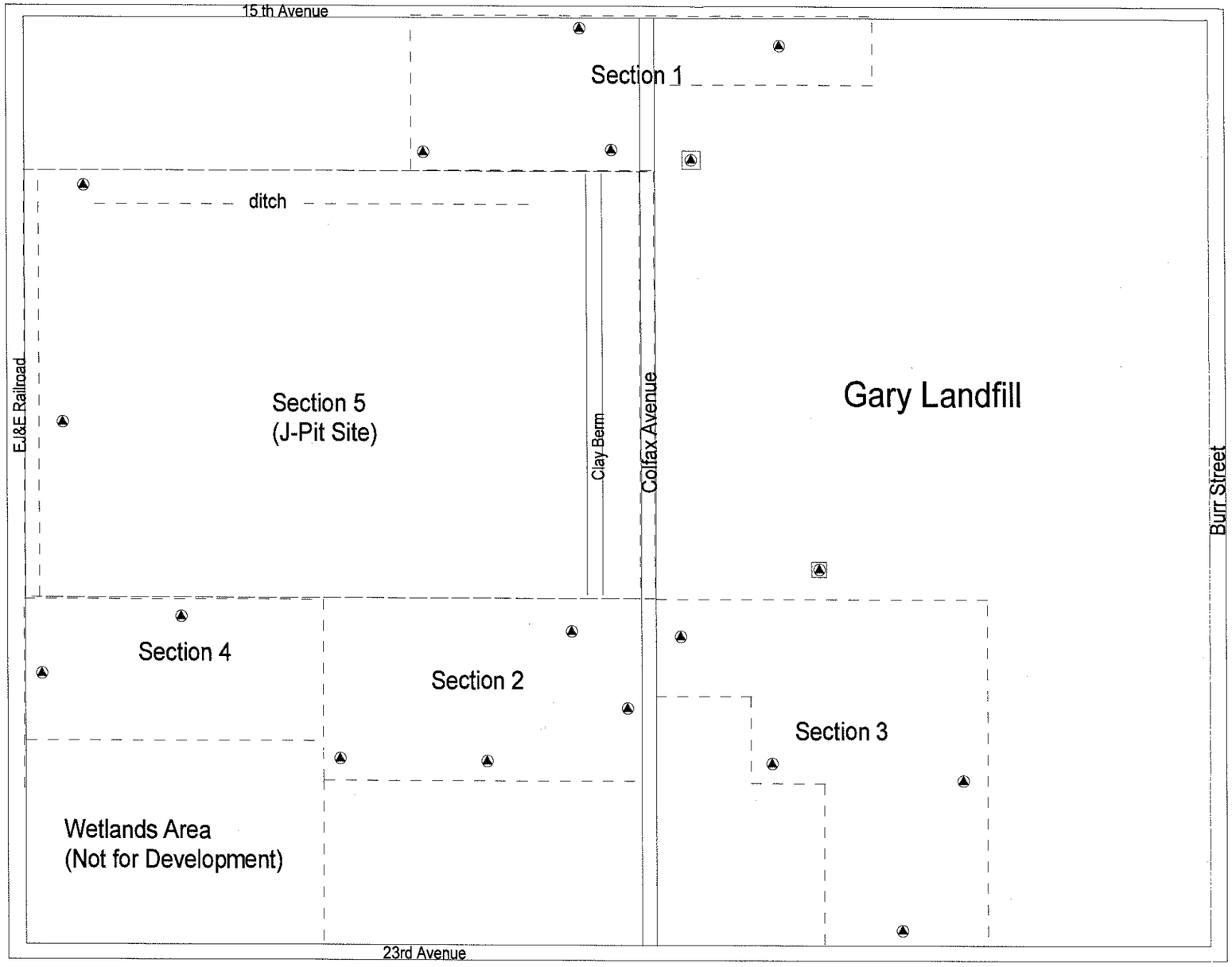
BAKER ENVIRONMENTAL, INC

**Figure 2-1**






| | | | | |
|---|---|---|---|---|
| **Drawn: RMS**<br>**Reviwed: JDP**<br>**5-02-02**<br>**Not to Scale** | **J-Pit Phase II Proposed Samplingl Location Map** | **BAKER ENVIRONMENTAL MERRILLVILLE, INDIANA** | **Figure 3-1** | Baker |




| | | | | |
|---|---|---|---|---|
| **Drawn: RMS**<br>**Reviwed: JDP**<br>**6-02-02**<br>**Not to Scale** | **J-Pit Phase II Proposed Monitoring Well Location Map** | **BAKER ENVIRONMENTAL MERRILLVILLE, INDIANA** | **Figure 3-2** | **Baker** |

# APPENDIX C

## TABLE OF CONTENTS

**FIELD OPERATIONS: Standard Operating Procedures**

# F101
# *BOREHOLE AND SAMPLE LOGGING*

## BOREHOLE AND SAMPLE LOGGING

### 1.0 PURPOSE

This SOP provides general reference information and technical guidance on borehole and sample logging. Borehole logs provide information that is used in the determination of geological conditions, assessment of contaminant distribution, and the evaluation of remedial actions.

### 2.0 SCOPE

This SOP provides descriptions of the standard techniques for borehole and sample logging. These techniques shall be used to provide consistent descriptions of subsurface lithology for each boring that is logged. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer may develop adequate classifications through careful, thorough observation and consistent application of the classification procedure.

### 3.0 DEFINITIONS

Soil classifications and terms are given in Sections 5.2 and 5.3. Rock classification and terms are presented in Section 5.4.

### 4.0 RESPONSIBILITIES

Project Manager – It is the responsibility of the Project Manager to ensure that field personnel responsible for borehole logging are familiar with these procedures. It also is the responsibility of the Project Manager to ensure that the appropriate documents (e.g., test boring logs, field logbooks, etc.) have been correctly and completely filled out by the drilling inspector.

Field Team Leader – The Field Team Leader is responsible for the overall supervision of the drilling and boring activities, and for ensuring that each borehole is completely logged by the responsible drilling inspector. The Field Team Leader also is responsible for ensuring that all drilling inspectors have been briefed on these procedures. The field team leader is responsible for providing copies of the test boring logs and field log books to the Project File via the Project Manager on a weekly basis, unless otherwise specified by the Project Manager.

Drilling Inspector – The drilling inspector (site geologist) is responsible for the direct supervision of boring and sampling activities. It is the Drilling Inspector's responsibility to log each boring, document subsurface conditions, complete the appropriate forms, and direct the drilling crew (or drilling supervisor).

### 5.0 PROCEDURES

The classification of soil and rock is one of the most important jobs of a drilling inspector or site geologist. It is imperative that the drilling inspector understand and accurately use the field classification system described in this SOP to maintain a consistent flow of information. This identification is based on both visual examination and manual tests. The results of the boring activities, including soil and rock classifications, shall be recorded on a Field Test Boring Record (Attachment A) or the field notebook.

## 5.1 Test Boring Record

Each boring shall be fully described in a Field Test Boring Record. The drilling inspector shall log the boring, as it is being drilled, by recording relevant data on the Boring Record. It may be more appropriate to record the boring information in a bound field log book so that all boring logs recorded (by each drilling inspector) are located in one source. The use of a field log book precludes the possibility of losing individual test boring log sheets. Furthermore, use of the field log book allows for the recording of additional information (i.e. notes) for which space is not allocated on the Field Test Boring Record. Field Test Boring Records may then be transcribed from the field log book, but must be completed at a minimum, on a weekly basis. The Field Test Boring Records must be completely filled out and signed prior to demobilization from the site. Field Test Boring Records must also be legible. Completed Field Test Boring Records shall be converted to report format using a Test Boring Record.

The data which is to be included on the Test Boring Records, when applicable is listed below.

1. Project name, location, and Project and Task Number.
2. Date(s).
3. Identifying number and location of each boring.
4. Soil classifications in accordance with the Unified Soil Classification System (see Section 5.2 and Attachment B). These classifications will be noted in the field by the drilling inspector and revised, if necessary, based on laboratory analysis and review. Both field determined USCS soil classification and a soil description shall be included on the Test Boring Record.
5. Depth limits, and the type and number of samples taken.
6. The number of blows required for each 6-inch penetration of a split-spoon sampler and for each 12-inch penetration of casing. The percentage of sample recovered, hammer weight, fall length, and hydraulic pressures to push thin-walled tubes.
7. Depth to water as first encountered during drilling operations, along with the method of determination. Any distinct water bearing zones shall also be delineated.
8. Loss of drilling fluid (indicative of subsurface voids) and the interval over which it was observed.
9. Identification of equipment used, including model and type of drilling rig, size of split spoon samplers, auger types and sizes, etc.
10. Start date and completion dates for the boring.
11. Name of the drilling company and the driller.
12. Size and length of the casing used in each hole.
13. Observations of visual contamination.
14. Field instrument readings (i.e., photoionization detector, organic vapor analyzer).

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## TEST BORING RECORD

PROJECT:

BORING NO.:

COORDINATES: EAST: _____ NORTH: _____

ELEVATION: SURFACE: _____ TOP OF PVC CASING: _____

| RIG: | SPLIT SPOON | CASING | AUGERS | CORE BARREL | DATE | PROGRESS (FT.) | WEATHER | WATER DEPTH (FT.) | TIME |
|---|---|---|---|---|---|---|---|---|---|
| SIZE (DIAM.) | | | | | | | | | |
| LENGTH | | | | | | | | | |
| TYPE | | | | | | | | | |
| HAMMER WT. | | | | | | | | | |
| FALL | | | | | | | | | |
| STICK UP | | | | | | | | | |

REMARKS:

**SAMPLE TYPE**

S = Split Spoon A = Auger
T = Shelby Tube W = Wash
R = Air Rotary C = Core
D = Denison P = Piston
N = No Sample

**DEFINITIONS**

SPT = Standard Penetration Test (ASTM D-1586)(Blows/0.5')
RQD = Rock Quality Designation (%)
Lab. Class. = USCS (ASTM D-2487) or AASHTO (ASTM D-3282)
Lab. Moist. = Moisture Content (ASTM D-2216) Dry Weight Basis

| Depth (ft.) | Samp. Type and No. | Samp. Rec. (ft. & %) | SPT or RQD | Lab Class. or Pen. Rate | PID (ppm) | Visual Description | Elevation |
|---|---|---|---|---|---|---|---|
| 1 | | | | | | | |
| 2 | | | | | | | |
| 3 | | | | | | | |
| 4 | | | | | | | |
| 5 | | | | | | | |
| 6 | | | | | | | |
| 7 | | | | | | | |
| 8 | | | | | | | |
| 9 | | | | | | | |
| 10 | | | | | | Match to Sheet 2 | |

DRILLING CO.: _____ BAKER REP.: _____

DRILLER: _____ BORING NO.: _____

TEST BORING RECORD

PROJECT: _____
BORING NO.: _____

| SAMPLE TYPE | | DEFINITIONS |
|---|---|---|
| S = Split Spoon | A = Auger | SPT = Standard Penetration Test (ASTM D-1586)(Blows/0.5') |
| T = Shelby Tube | W = Wash | RQD = Rock Quality Designation (%) |
| R = Air Rotary | C = Core | Lab. Class. = USCS (ASTM D-2487) or AASHTO (ASTM D-3282) |
| D = Denison | P = Piston | Lab. Moist. = Moisture Content (ASTM D-2216) Dry Weight Basis |
| N = No Sample | | |

| Depth (ft.) | Samp. Type and No. | Samp. Rec. (ft. & %) | SPT or RQD | Lab Class. or Pen. Rate | PID (ppm) | Visual Description | Elevation |
|---|---|---|---|---|---|---|---|
| 11 | | | | | | Continued from Sheet 1 | |
| 12 | | | | | | | |
| 13 | | | | | | | |
| 14 | | | | | | | |
| 15 | | | | | | | |
| 16 | | | | | | | |
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| 27 | | | | | | | |
| 28 | | | | | | | |
| 29 | | | | | | | |
| 30 | | | | | | | |

DRILLING CO.: _____
DRILLER: _____

BAKER REP.: _____
BORING NO.: _____

As the boring is advanced, the inspector shall evaluate the samples and the cuttings to determine the location of each stratigraphic unit. The descriptions should contain color, grain-size distribution, consistency, moisture, etc., in addition to the USCS classification category (Section 5.3.7).

Each sample collected for chemical or geotechnical analysis shall be handled as described in SOP F102.

## 5.2 Soil Classification

The data shall be recorded on a Field Test Boring Record, or in a field logbook. The method of deriving the classification should be described, or reference made to this SOP or other applicable manuals. Both the soil classification and the soil descriptions must be entered on the Field Test Boring Record. If required, the soil classification shall consist of the two-letter USCS classification; the soil description shall be much more detailed.

Where required, soils will be classified according to the USCS. The USCS method of classification is detailed in Attachment B and identifies soil types on the basis of grain-size and liquid limits, and categorizes them through the use of two letters. Although some laboratory testing is required for full USCS classification, preliminary classifications may be made in the field.

Fine-grained soils are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition; peat is designated by "Pt." Coarse-grained soils are divided into sand (S) or gravel (G). The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

The second letter of the two-letter USCS symbol provides information about the grain size distribution of granular soil, or the plasticity characteristics of fine-grained soils. These second letter modifiers are (P) poorly graded/well sorted, (W) well graded/poorly sorted, (C) clayey, (M) silty, (L) low plasticity, or (H) high plasticity.

## 5.3 Soil Descriptions

The Test Boring Records shall contain complete soil descriptions in addition to the two-letter USCS classification, if required. Soil descriptions include the following components: grain size identification with descriptive terms indicating the relative percentage of each grain size, color, consistency or relative density, moisture content, organic content, plasticity, and other pertinent observations such as visual contamination, HNu measurements, etc. A summary of the soil description components is given in Attachment C.

### 5.3.1 Grain Size Identification

In nature, soils are comprised of varying size, shape, and combinations of the various grain types. The following terms are used to indicate soil grain size:

| Size | Size Limits |
|---|---|
| Cobbles | 3-inches to 12-inches |
| Coarse gravel | 3/4-inches to 3-inches |
| Fine gravel | 4.76 mm (# 4 sieve size) to 3/4-inches |
| Coarse sand | 2 mm (# 10 sieve size) to 4.76 mm |
| Medium sand | 0.42 mm (# 40 sieve size) to 2 mm |
| Fine sand | 0.074 mm (# 200 sieve size) to 0.42 mm |
| Silt | 0.002 mm to 0.074 mm |
| Clay | less than 0.002 mm |

The proportion of each grain size (by weight percent) is indicated using the descriptive terms:

| | |
|---|---|
| Trace | 0 to 10 percent |
| Little | 10 to 20 percent |
| Some | 20 to 35 percent |
| And (or an adjective form of the grain size, i.e., sandy, silty, clayey) | 35 to 50 percent |

Some examples of soil grain size descriptions are:

- Silty fine sand: 50 to 65 percent fine sand and 35 to 50 percent silt.
- Medium to coarse sand, some silt: 65 to 90 percent medium to coarse sand, 20 to 35 percent silt.
- Fine sandy silt, trace clay: 50 to 65 percent silt, 35 to 50 percent fine sand, and 0 to 10 percent clay.

The soil type may be classified as noncohesive, granular soils or as cohesive, fine-grained soils as discussed in Section 5.3.3. The grain shape of a soil usually does not need to be determined unless unusual or unique features are readily apparent.

### 5.3.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light-gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent between borings.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors because sampling devices tend to smear the sample surface creating color variations between interior and exterior.

The term "mottled" shall be used to indicate soil irregularly marked with spots of different colors. Soil color charts shall not be used unless specified by the Project Manager.

### 5.3.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the drilling inspector first shall identify the soil type. Granular soils contain predominantly sands and gravels. These types of soil are noncohesive (particles do not adhere well when compressed). Conversely, fine-grained soils which contain predominantly silts and clays are cohesive (particles will adhere when compressed).

The density of noncohesive, granular soils or the consistency of cohesive soils is classified according to standard penetration resistances obtained from split-spoon (split-barrel) sampling performed according to ASTM D-1586. Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12-inches into the material using a 140-pound hammer falling freely through 30-inches. In cases where geotechnical information is required, the standard penetration test is performed by driving the sampler through an 18-inch sample interval, the number of blows will then be recorded for each six-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of the sample interval. It is important to note that if gravel and rock fragments are broken by the sampler, or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This must be noted on the Field Test Boring Record and referenced to the sample number. In cases where soil sampling for environmental analytical analysis is required, 24-inch spoon barrels can be used in order to obtain a sufficient quantity of sample for required analysis. Accordingly, the second and third 6-inch increments will be used to calculate the relative density.

The relative density designations for noncohesive soils are:

| Designation | Standard Penetration Resistance (Blows per Foot) |
|---|---|
| Very loose | Less than 4 |
| Loose | 4 to 10 |
| Medium dense | 10 to 30 |
| Dense | 30 to 50 |
| Very dense | Greater than 50 |

The consistency of cohesive soils is also determined by blow counts as shown:

| Designation | Standard Penetration Resistance (Blows per Foot) |
|---|---|
| Very Soft | Less than 2 |
| Soft | 2 to 4 |
| Medium Stiff | 4 to 8 |
| Stiff | 8 to 15 |
| Very Stiff | 15 to 30 |
| Hard | Over 30 |

### 5.3.4 Moisture Content

Moisture content is estimated in the field according to four categories: dry, damp, moist, and wet:

| Designation | Moisture Content |
|---|---|
| Dry | 0 to 10 percent |
| Damp | 10 to 20 percent |
| Moist | 20 to 35 percent |
| Wet | 35 to 50 percent |

Little or no water should appear in dry soil. Wet soils appear to contain all the water they can possibly hold (i.e., saturated). Damp and moist are subjective. Laboratory analysis should be performed if it is necessary to accurately determine the natural water content.

### 5.3.5 Stratification

Stratification can only be determined after the split-barrel sampler is opened. Typically, bedding thicknesses are described as follows:

| Designation | Bedding Spacing |
|---|---|
| Indistinct | No bedding apparent |
| Laminated | Less than 1/2-inch |
| Very thin | 1/2-inch to 1-inch |
| Thin | 1-inch to 4-inches |
| Medium | 4-inches to 1-foot |
| Thick | 1-foot to 3-feet |
| Massive | Greater than three feet |

### 5.3.6 Texture/Fabric/Bedding

The texture/fabric/bedding of a soil shall be described, where appropriate. Texture is described as the relative angularity of the soil particles: rounded, subrounded, subangular, angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation or orientation. The bedding structure also shall be noted (e.g., stratified, lensatic, nonstratified, heterogeneous varved, etc.).

### 5.3.7 Summary of Soil Descriptions

In summary, soils shall be classified in a similar manner by each drilling inspector. The soil description shall include:

- Soil grain size with appropriate descriptors
- Color
- Relative density and/or consistency
- Moisture content
- Stratification
- Texture/fabric/bedding
- Other distinguishing features

These descriptors are evaluated and the soil classified according to the USCS. All information, measurements and observations shall be legibly recorded on a Field Test Boring Record.

## 5.4 Sedimentary Rock Classifications

Rocks are grouped into three main divisions: sedimentary, igneous, and metamorphic. Sedimentary rocks are the most predominant type exposed at the earth's surface. As such, this section will consider only classification of sedimentary rocks. Standard geologic references should be used for the complete classification of sedimentary, igneous and metamorphic rocks.

For the purpose of completing the Field Test Boring Record in the field, sedimentary rocks should be classified using the following hierarchy:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Rock Quality Designation
- Weathering
- Other characteristics

### 5.4.1 Rock Type

There are numerous types of sedimentary rocks such as sandstone, shale, siltstone, claystone, conglomerate, limestone, dolomite, coal, etc. The drilling inspector should select the most appropriate rock type based on experience. Some of the references listed in Section 7.0 provide a more complete discussion of sedimentary rock types.

In addition to selecting a rock type, the drilling inspector should record the grain size (and composition of grains and cement, if apparent) on the Field Test Boring Record. The following designation should be used to describe grain size in sedimentary rocks:

| Designation | Grain Size Diameter |
|---|---|
| Cobbles | Greater than 64 mm (2.5-inches) |
| Pebbles | 4 mm (0.16-inches) to 64 mm |
| Granules | 2 mm (0.08-inches) to 4 mm |
| Very Coarse Sand | 1 mm to 2 mm |
| Coarse Sand | 0.5 mm to 1 mm |
| Medium Sand | 0.25 mm to 0.5 mm |
| Fine Sand | 0.125 mm to 0.25 mm |
| Very Fine Sand | 0.0625 mm to 0.125 mm |
| Silt | 0.0039 mm to 0.0625 mm |
| Clay | Smaller than 0.0039 mm |

For individual boundaries of grain size, a scale can be used for coarse-grained rocks. However, the division between silt and clay likely will not be measurable in the field. This boundary shall be determined by use of a hand lens. If the grains cannot be seen with the unaided eye, but are distinguishable with a hand lens (5x magnification) the sample is silt. If the grains are not distinguishable with a hand lens, the sample is clay.

### 5.4.2 Color

The color of rock can be determined in a manner similar to that for soil samples. Rock cores or fragments shall be classified while wet, when possible. Rock color charts shall not be used unless specified by the Project Manager.

### 5.4.3 Bedding Thickness

The bedding thickness designation for soils (Section 5.3.5) shall also be used for rock descriptions.

### 5.4.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness follows:

- Very Soft - Very soft indicates that the rock is easily gouged by a knife, easily scratched by a fingernail, and/or easily broken by hand
- Soft - Soft indicates that the rock may be gouged by a knife, scratched by a fingernail, difficult to break by hand, and/or powders when hit by a hammer.
- Medium Hard - Medium hard indicates that the rock is easily scratched by a knife and/or is easily broken when hit by a hammer.
- Hard - Hard indicates that the rock is difficult to scratch with a knife but may be broken with a hammer.
- Very Hard - Very hard indicates that the rock is difficult to break with a hammer.

Note the difference in usage between the words "scratch" and "gouge." A scratch shall be considered a slight depression in the rock while a gouge is much deeper.

### 5.4.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is measured and is described by the following terms:

- Very Broken - Less than a 2-inch spacing between fractures
- Broken - A 2-inch to 1-foot spacing between fractures
- Blocky - A 1-foot to 3-foot spacing between fractures
- Massive - A 3-foot to 10-foot spacing between fractures

### 5.4.6 Rock Quality Designation

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding four inches and dividing by the total length of core run:

$$RQD\ (\%) = r/l \times 100$$

Where:

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches, and have resulted from natural breaks. Natural breaks include slickenslides, joints, compaction slicks, bedding plane partings (not caused by drilling) friable zones, etc.

l = Total length of core run.

The results of the RQD calculations shall be recorded on the Field Test Boring Record.

### 5.4.7 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and also is useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Decomposed - Soft to very soft, bedding and fractures indistinct, no cementation.
- Highly weathered - very soft to soft, with medium hard relic rock fragments, little to moderate cementation. Vugs and openings in bedding and fracture planes, some of which may be filled.
- Weathered - Soft to medium hard. Good cementation, bedding and fractures are pronounced. Uniformly stained.
- Slightly weathered - Medium hard. Fractures pronounced, nonuniform staining, bedding distinct.
- Fresh - Medium hard to hard. No staining. Fractures may be present, bedding may or may not be distinct.

### 5.4.8 Other Characteristics

The following items should be included in rock description, where applicable:

- Description of contacts between rock units (sharp or gradational)
- Stratification
- Description of any filled cavities
- Cementation (calcareous, siliceous, hematitic, etc.)
- Description of joints and open fractures (with strike and dip, if possible)
- Observation of the presence of fossils

### 5.4.9 Additional Terms

The following terms also are used to further identify rocks:

- Seam - thin (12-inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of an accessory material.
- Few - Indicates insignificant (0 to 15 percent) amounts of an accessory material.
- Interbedded - Indicates thin or very thin alternating seams of material occurring in approximately equal amounts.
- Interlayered - Indicates thick alternating seams of material occurring in approximately equal amounts.

## 6.0 QUALITY ASSURANCE RECORDS

Quality Assurance Records shall consist of completed Field Test Boring Records and Test Boring Records.

## 7.0 REFERENCES

1. American Society for Testing and Materials, 1990. Standard Methods for Classification of Soils for Engineering Purposes. ASTM Method D2487-90, Annual Book of Standards, ASTM, Philadelphia, Pennsylvania.

2. American Society for Testing and Materials, 1990. Standard Practice for Description and Identification of Soils (Visual - Manual Procedure). ASTM Method D2488-90, Annual Book of Standards, ASTM, Philadelphia, Pennsylvania.

**ATTACHMENT A**

**EXAMPLE TEST BORING RECORD**

# TEST BORING RECORD

PROJECT: Building P-64
S.O. NO.: 19010-51-SRN
COORDINATES: EAST: _____ NORTH: _____
ELEVATION: SURFACE: _____ TOP OF PVC CASING: _____
BORING NO.: B-1

RIG: Mobile B-57

| | SPLIT SPOON | CASING | AUGERS | CORE BARREL | DATE | PROGRESS (FT) | WEATHER | WATER DEPTH (FT) | TIME |
|---|---|---|---|---|---|---|---|---|---|
| SIZE (DIAM.) | 1-3/8"ID | | 6-1/4" ID | | 5/31/91 | 14.0 | Sunny, 80°-90°F | — | — |
| LENGTH | 2.0' | | 5.0' | | | | | | |
| TYPE | STD. | | HSA | | | | | | |
| HAMMER WT. | 140# | | | | | | | | |
| FALL | 30" | | | | | | | | |
| STICK UP | | | | | | | | | |

REMARKS: Advanced boring to 14 ft. taking continuous 2-foot split-spoon samples; no monitoring well installed - borehole grouted to surface.

SAMPLE TYPE
S = Split Spoon A = Auger
T = Shelby Tube W = Wash
R = Air Rotary C = Core
D = Denison P = Piston
N = No Sample

DEFINITIONS
SPT = Standard Penetration Test (ASTM D-1586) (Blows/0.5')
RQD = Rock Quality Designation (%)
Lab Class. = USCS (ASTM D-2487) or AASHTO (ASTM D-3282)
Lab Moist. = Moisture Content (ASTM D-2216) Dry Weight Basis

| Depth (Ft.) | Sample Type and No. | Samp. Rec. Ft. & % | SPT or RQD | Lab. Class. or Pen. Rate | PID (ppm) | Visual Description | Elevation |
|---|---|---|---|---|---|---|---|
| 0 | | | | | | TOPSOIL, grass roots; tan, gray; medium dense; dry | |
| 1.0 | | | | | | | 1.0' |
| 2.0 | S-1 | 1.3/2.0 65% | 3 7 9 5 | | 0 | SAND, fine- grained, trace gravel, trace silt; tan, brown; loose; moist to damp | |
| 4.0 | S-2 | 1.3/2.0 65% | 4 3 4 4 | | 0 | SAND, fine- grained, trace silt, trace grass roots; tan, brown; loose; moist to damp | 4.0' |
| 6.0 | S-3 | 2.0/2.0 100% | 11 12 10 8 | | 0 | SAND, fine to medium-grained, trace silt; tan, brown, orange; medium dense; moist to wet; water table at 6.0' | |
| 7.0 | | | | | | | 7.0' |
| 8.0 | S-4 | 1.8/2.0 90% | 3 4 3 4 | | 0 | SAND, medium to coarse-grained, trace silt; tan, gray, orange; loose; wet | |
| 9.0 | | | | | | | 9.0' |
| 10.0 | S-5 | 2.0/2.0 100% | 1 0 1 1 | | 0 | SAND, medium-grained, trace silt; gray, orange; very loose; wet | |
| | | | | | | Match to Sheet 2 | |

DRILLING CO.: ATEC Associates
DRILLER: M. Miller
BAKER REP.: R. Bonelli
BORING NO.: B-1

# ATTACHMENT B

# UNIFIED SOIL CLASSIFICATION SYSTEM

| MAJOR DIVISIONS | | | GROUP SYMBOLS | TYPICAL NAMES |
|---|---|---|---|---|
| COARSE-GRAINED SOILS | GRAVELS | CLEAN GRAVELS | GW | Well-Graded Gravels and Gravel-Sand Mixtures, Little or No Fines |
| | | | GP | Poorly Graded Gravels and Gravel-Sand Mixtures, Little or No Fines |
| | | GRAVELS WITH FINES | GM | Silty Gravels, Gravel-Sand-Silt Mixtures |
| | | | GC | Clayey Gravels, Gravel-Sand-Clay Mixtures |
| | SANDS | CLEAN SANDS | SW | Well-Graded Sands and Gravelly Sands, Little or No Fines |
| | | | SP | Poorly Graded Sands and Gravelly Sands, Little or No Fines |
| | | SANDS WITH FINES | SM | Silty Sands, Sand-Silt Mixtures |
| | | | SC | Clayey Sands, Sand-Clay Mixtures |
| FINE-GRAINED SOILS | SILTS AND CLAYS LIQUID LIMIT >50% | | ML | Inorganic Silts, Very Fine Sands, Rock Flour, Silty or Clayey Fine Sands |
| | | | CL | Inorganic Clays of Low to Medium Plasticity, Gravelly Clay, Sandy Clays, Silty Clays, Lean Clays |
| | | | OL | Organic Silts and Organic Silty Silty Clays of Low Plasticity |
| | SILTS AND CLAYS LIQUID LIMIT <50% | | MH | Inorganic Silts, Micaceous or Diatomaceous Fine Sands or Silts, Elastic Silts |
| | | | CH | Inorganic Clays of High Plasticity, Fat Clays |
| | | | OH | Organic Clays of Medium to High Plasticity |
| HIGHLY ORGANIC SOILS | | | PT | Peat, Muck and Other Highly Organic Soils |

## ASTM GEOLOGICAL DESCRIPTION CHECKLIST FOR FINE-GRAINED AND PARTLY ORGANIC SOILS

1. TYPICAL NAME: Sandy Silt, Silt; Silty Clay, Clay; Clayey Silt, Sandy Clay; Organic Silt, Organic Clay
2. MAXIMUM PARTICLE SIZE
3. SIZE DISTRIBUTION
4. DRY STRENGTH: None, Very Low, Low, Medium, High, Very High
5. DILATENCY: None, Slow, Rapid
6. PLASTIC THREAD: Weak and Soft, Medium, Stiff, Very Stiff
7. PLASTICITY OF FINES: None, Low, Medium, High
8. COLOR: Use Munsell Notation, If Possible
9. ODOR: None, Earthy, Organic
10. MOISTURE CONTENT: Dry, Moist, Wet, Saturated
11. CONSISTENCY: Soft, Firm (Medium), Stiff, Very Stiff, Hard
12. STRUCTURE: Stratified, Laminated, Fissured, Slickensided, Blocky Lensed, Homogeneous
13. CEMENTATION: Weak, Strong
14. LOCAL OR GEOLOGIC NAME

## ASTM GEOLOGICAL DESCRIPTION CHECKLIST FOR COARSE GRAINED SOILS

1. TYPICAL NAME: Boulder, Cobble, Gravel, Sand (Add Descriptive Adjectives For Minor Constituents)
2. GRADUATION: Well Graded, Poorly Graded
3. MAXIMUM PARTICLE SIZE
4. SIZE DISTRIBUTION: Percent Gravel, Sand and Fines
5. GRAIN SHAPE: Angular, Subangular, Subrounded, Rounded
6. MINERALOGY: Rock Type For Gravel, Predominant Mineral in Sand
7. COLOR: Use Munsell Notation, If Possible
8. ODOR: None, Earthy, Organic
9. MOISTURE CONTENT: Dry, Moist, Wet, Saturated
10. NATURAL DENSITY: Loose Dense
11. STRUCTURE: Stratified, Lensed, Nonstratified
12. CEMENTATION: Weak, Strong
13. LOCAL OR GEOLOGIC NAME

NOTES

# ATTACHMENT C

# SOIL AND ROCK DESCRIPTION SUMMARY

## SOIL DESCRIPTION

### GRAIN SIZE IDENTIFICATION

| NAME | SIZE LIMITS |
|---|---|
| Boulder | 12" OR MORE |
| Cobbles | 3" - 12" |
| Coarse Gravel | 3/4" - 3" |
| Fine Gravel | 4.76 mm (#4) - 3/4" |
| Coarse Sand | 2 mm (#10) - 4.76 mm (#4) |
| Medium Sand | 0.42 mm (#40) - 2 mm (#10) |
| Fine Sand | 0.074 mm (#200)-0.42 mm (#40) |
| Silt | 0.002 mm-0.074 mm (#200) |
| Clay | Less than 0.002 mm |

### RELATIVE DENSITY

#### NONCOHESIVE SOIL

| TERM | SPT (Blows/ft) |
|---|---|
| Very Loose | Below 4 |
| Loose | 4-10 |
| Medium Dense | 10-30 |
| Dense | 30-50 |
| Very Dense | OVER 50 |

#### COHESIVE SOILS

| TERM | SPT (Blows/ft) |
|---|---|
| Very Soft | BELOW 2 |
| Soft | 2-4 |
| Medium Stiff | 4-8 |
| Stiff | 8-15 |
| Very Stiff | 15-30 |
| Hard | OVER 30 |

| MOISTURE | DESCRIPTIVE TERMS | |
|---|---|---|
| Dry - | Trace | 0-10% |
| Damp | Little | 10-20% |
| Moist | Some | 20-35% |
| Wet | And | 35-50% |

## ROCK DESCRIPTIONS

### HARDNESS

| | |
|---|---|
| Very Soft - | Easily gouged by knife, easily scratched by fingernail, easily broken by hand |
| Soft - | Gouged by knife, scratched by fingernail, difficult to break by hand, powders with hammer |
| Medium Hard - | Easily scratched by knife, easily broken with hammer |
| Hard - | Difficult to scratch, breaks with hammer |
| Very Hard - | Difficult to break, rings when struck |

### WEATHERING

| | | |
|---|---|---|
| Decomposed | - | Soft to Very soft, bedding and fractures indistinct, no cementation. |
| Highly Weathered | - | Very soft to soft, with medium hard relict rock fragments; little to moderate cementation. Vugs, openings in bedding and fractures (may be filled). |
| Weathered | - | Soft to medium hard. Good cementation, bedding and fractures are pronounced. Uniformly stained. |
| Slightly Weathered | - | Medium hard. Fractures pronounced, non-uniform staining, bedding distinct. |
| Fresh | - | Medium hard to hard. No staining. Fractures may be present. Bedding may or may not be indistinct. |

### BEDDING AND FRACTURES:

| SPACING | BEDDING | FRACTURES |
|---|---|---|
| | Indistinct | |
| LESS THAN 1/2" (1 cm) | Laminated | Fissile |
| 1/2" to 1" (1cm-3cm) | Very Thin | Very Close |
| 1" TO 4" (3cm-10cm) | Thin | Close |
| 4" TO 1' (10cm-30cm) | Moderate | Moderate |
| 1' TO 3' (30 cm-1m) | Thick | Wide |
| 3' TO 10' (1m-3m) | Massive | Very Wide |

### CONTACTS:

_____ = DEFINITE

_ _ _ _ _ = INDEFINITE

. . . . . = GRADATIONAL

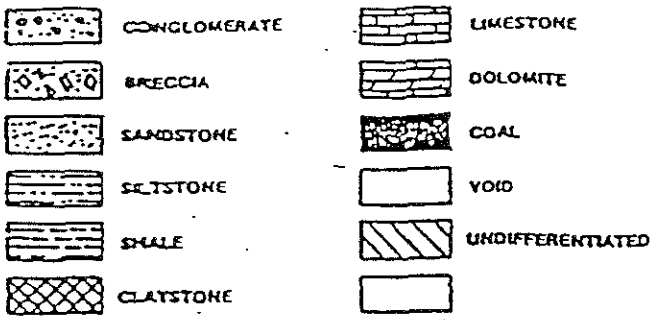
### SAMPLE TYPE

S = Split Spoon
T = Shelby Tube
R = Air Rotary
D = Denison
A = Auger
W = Wash (Roller Bit)
C = Core
P = Piston
N = No Sample Taken

### ABBREVIATIONS

HS = Hollow Stem
NP = Non Plastic
-PL = Below the Plastic Limit
PL = At the Plastic Limit
+PL = Above the Plastic Limit
+LL = Above the Liquid Limit
SPT = Standard Penetration Test
RQD = Rock Quality Designation

## ROCK SYMBOLS




## HARDNESS

V. SOFT - CORE RECOVERY < 50%, EASILY GOUGED BY KNIFE OR SCREWDRIVER, EASILY SCRATCHED BY FINGERNAIL, EASILY BROKEN BY HAND

SOFT - CORE RECOVERY 50 - 75%, GOUGED BY KNIFE OR SCREWDRIVER, SCRATCHED BY FINGERNAIL, DIFFICULT TO BREAK BY HAND, POWDERS w/HAMMER

MED. HD. - CORE RECOVERY > 75%, EASILY SCRATCHED BY KNIFE OR SCREWDRIVER, EASILY BROKEN BY HAMMER

HD. - DIFFICULT TO SCRATCH, BREAKS w/HAMMER

V. HD. - DIFFICULT TO BREAK, RINGS WHEN STRUCK

| SPACING | BEDDING | FRACTURES |
|---|---|---|
| | INDISTINCT | |
| LESS THAN ½" (1cm) | LAMINATED | FISSILE |
| ½" To 1" (1cm-3cm) | VERY THIN | VERY CLOSE |
| 1" To 4" (3cm-10cm) | THIN | CLOSE |
| 4" To 1' (10cm-30cm) | MODERATE | MODERATE |
| 1' To 3' (30cm-1m) | THICK | WIDE |
| 3' To 10' (1m-3m) | MASSIVE | VERY WIDE |

## WEATHERING

DECOMPOSED - SOFT - V. SOFT, BEDDING AND FRACTURES INDISTINCT, NO CEMENTATION

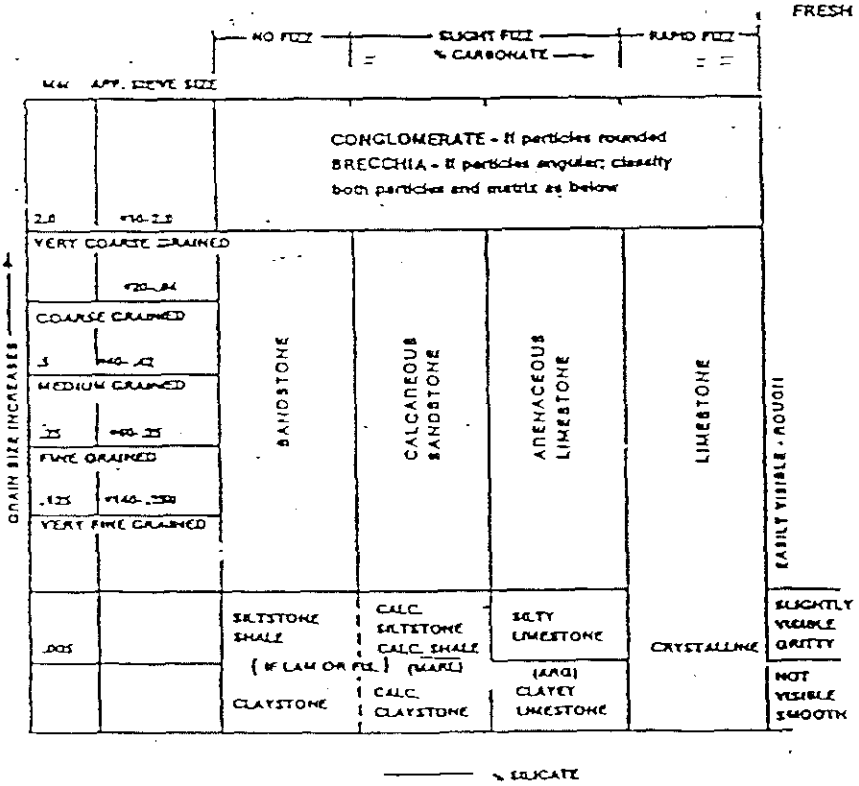
HL. WTHR. - V. SOFT - SOFT, w/MED. HD. RELICT ROCK FRAGMENTS; LITTLE TO MOD. CEMENTATION, VUGS, OPENINGS IN BEDDING AND FRACTURES (MAY BE CLAY OR CALC. FILLED)

WTHR. - SOFT TO MED. HD., GOOD CEMENTATION, BEDDING AND FRACTURES ARE PRONOUNCED, UNIFORMLY STAINED

SL. WTHR. - MED. HD., FRACTURES PRONOUNCED, NON-UNIFORM STAINING, BEDDING DISTINCT

FRESH - MED. HD. TO HD., NO STAINING, FRACTURES MAY BE PRESENT, BEDDING MAY OR MAY NOT BE DISTINCT

## COMMON LOCAL SEDIMENTARY ROCK CLASSIFICATIONS




RQD = $\frac{L}{R}$ - NTYPE CORE ONLY

L = TOTAL LENGTH IN A RUN OF CORE PIECES LONGER THAN 4"

R = LENGTH OF THE RUN

## ROCK DESCRIPTIONS

Designation: D 2488 – 90



# Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.*

## 1. Scope

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (See Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.5 The values stated in inch-pound units are to be regarded as the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Test Method for Classification of Soils for Engineering Purposes[2]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

## 3. Terminology

3.1 *Definitions:*

3.1.1 Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:

*Cobbles*—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and

*Boulders*—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1.2 *clay*—soil passing a No. 200 (75-μm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.1.3 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

*coarse*—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

*fine*—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.1.4 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.1.5 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.1.6 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.1.7 *sand*—particles of rock that will pass a No. 4

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[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

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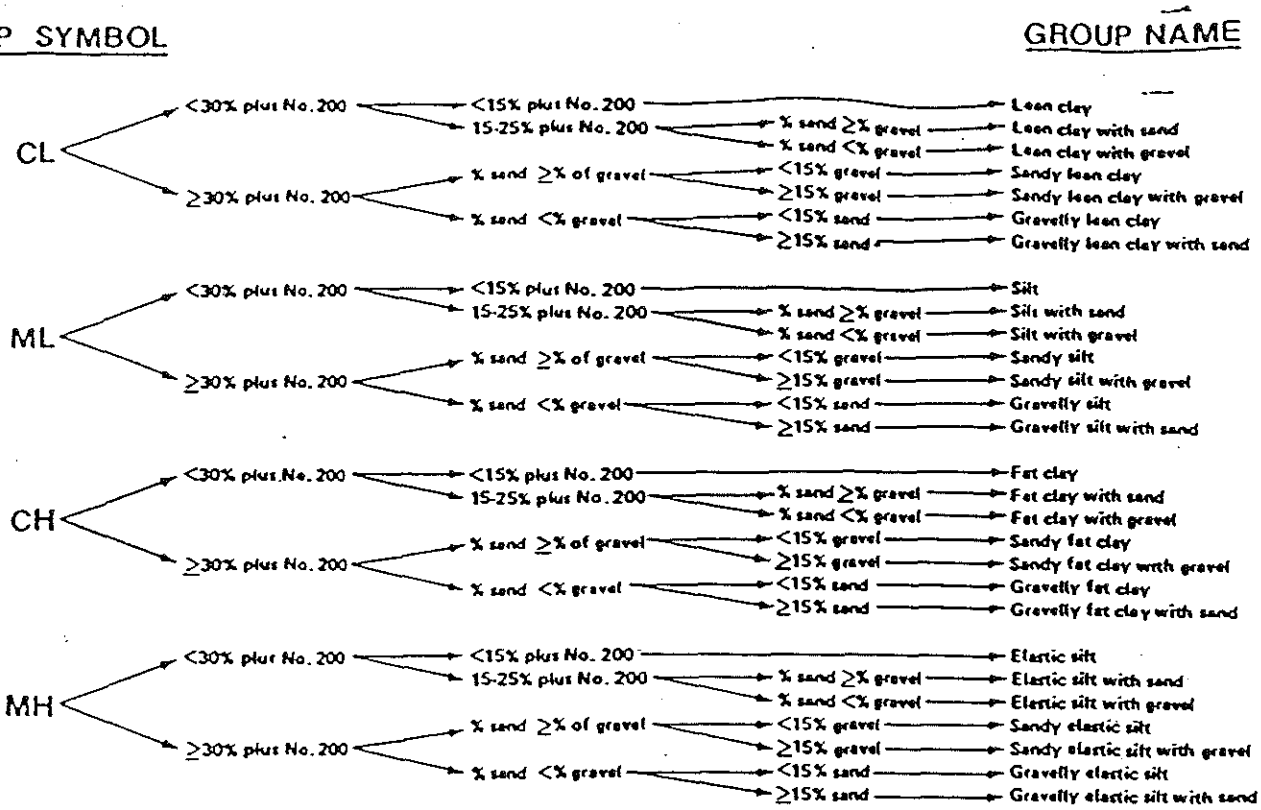
[2] *Annual Book of ASTM Standards*, Vol 04.08.




OTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)

75-mm) sieve and be retained on a No. 200 (75-μm) sieve h the following subdivisions:

*oarse*—passes a No. 4 (4.75-mm) sieve and is retained on Jo. 10 (2.00-mm) sieve.

*nedium*—passes a No. 10 (2.00-mm) sieve and is retained a No. 40 (425-μm) sieve.

*ine*—passes a No. 40 (425-μm) sieve and is retained on a . 200 (75-μm) sieve.

3.1.1.8 *silt*—soil passing a No. 200 (75-μm) sieve that is nplastic or very slightly plastic and that exhibits little or no ength when air dry. For classification, a silt is a fine-ained soil, or the fine-grained portion of a soil, with a isticity index less than 4, or the plot of plasticity index sus liquid limit falls below the "A" line (see Fig. 3 of Test ethod D 2487).

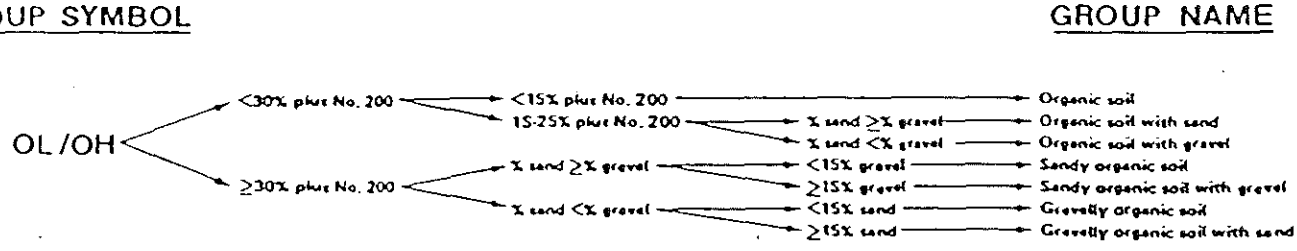
## 4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Figs. 1a and 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

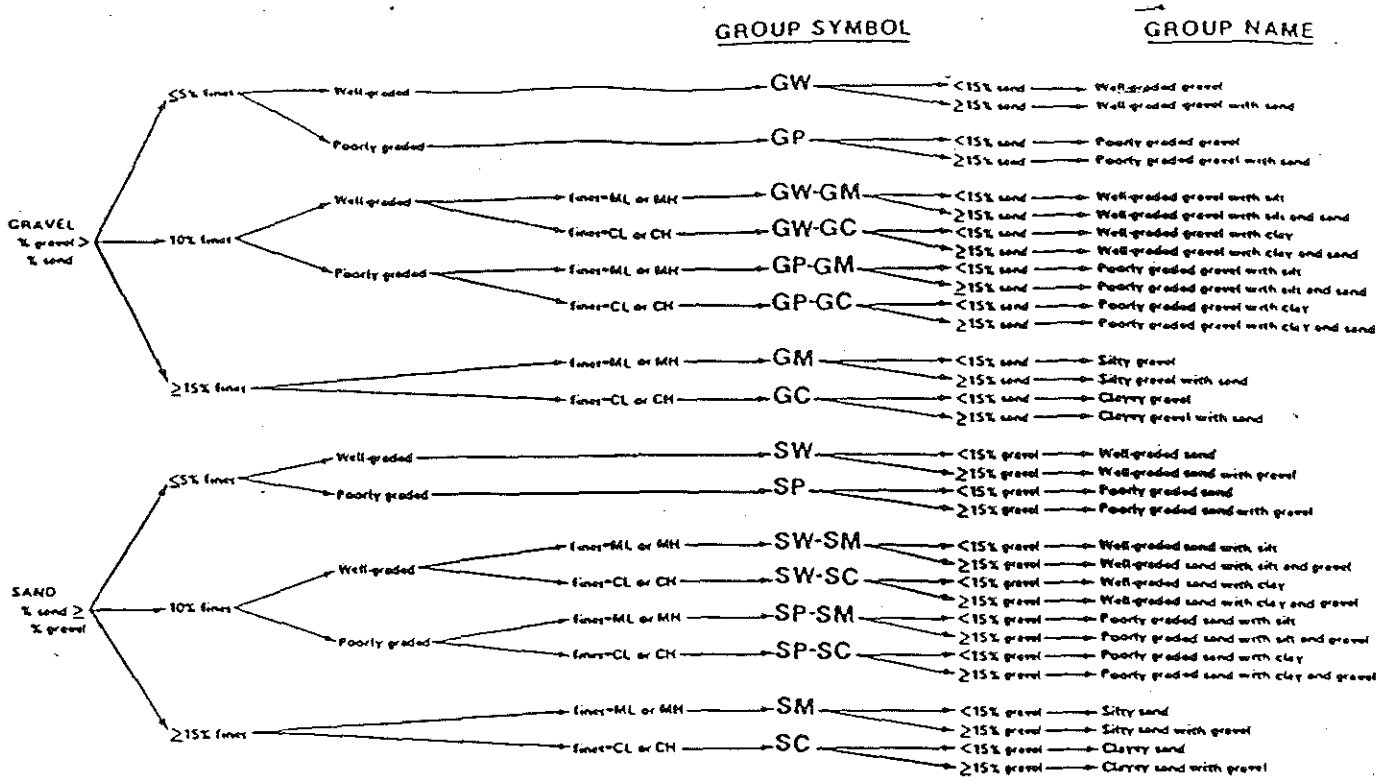
NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols*.

*Dual Symbol*—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or




OTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)




NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

*Borderline Symbol*—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

## 5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together, one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

## 6. Apparatus

6.1 *Required Apparatus:*

6.1.1 *Pocket Knife or Small Spatula.*

6.2 *Useful Auxiliary Apparatus:*

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens.*

## 7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

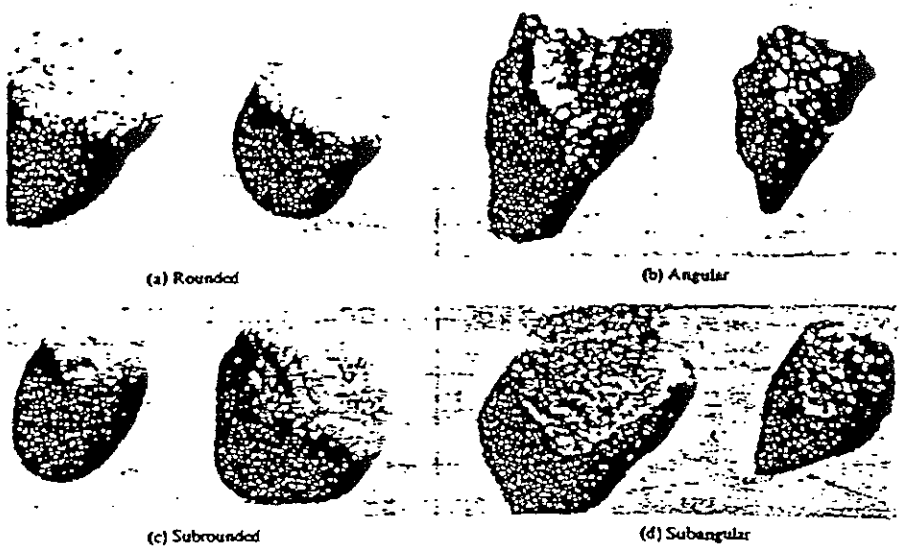



FIG. 3 Typical Angularity of Bulky Grains

## Safety Precautions

8.1 When preparing the dilute HCl solution of one part ncentrated hydrochloric acid (10 *N*) to three parts of :tilled water, slowly add acid into water following necessary ety precautions. Handle with caution and store safely. If ion comes into contact with the skin, rinse thoroughly water.

8.2 Caution—Do not add water to acid.

## Sampling

9.1 The sample shall be considered to be representative of : stratum from which it was obtained by an appropriate, cepted, or standard procedure.

NOTE 5—Preferably, the sampling procedure should be identified as ing been conducted in accordance with Practices D 1452, D 1587, or 2113, or Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 6—Remarks as to the origin may take the form of a boring mber and sample number in conjunction with a job number, a logic stratum, a pedologic horizon or a location description with pect to a permanent monument, a grid system or a station number l offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minum amount of the specimen to be examined shall be in accordance with the following schedule:

| Maximum Particle Size, Sieve Opening | Minimum Specimen Size, Dry Weight |
|---|---|
| 4.75 mm (No. 4) | 100 g (0.5 lb) |
| 9.5 mm (⅜ in.) | 200 g (0.5 lb) |
| 19.0 mm (¾ in.) | 1.0 kg (2.2 lb) |
| 38.1 mm (1½ in.) | 8.0 kg (18 lb) |
| 75.0 mm (3 in.) | 60.0 kg (132 lb) |

NOTE 7—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

| Description | Criteria |
|---|---|
| Angular | Particles have sharp edges and relatively plane sides with unpolished surfaces |
| Subangular | Particles are similar to angular description but have rounded edges |
| Subrounded | Particles have nearly plane sides but have well-rounded corners and edges |
| Rounded | Particles have smoothly curved sides and no edges |

## 10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.

| | |
|---|---|
| Flat | Particles with width/thickness > 3 |
| Elongated | Particles with length/width > 3 |
| Flat and elongated | Particles meet criteria for both flat and elongated |

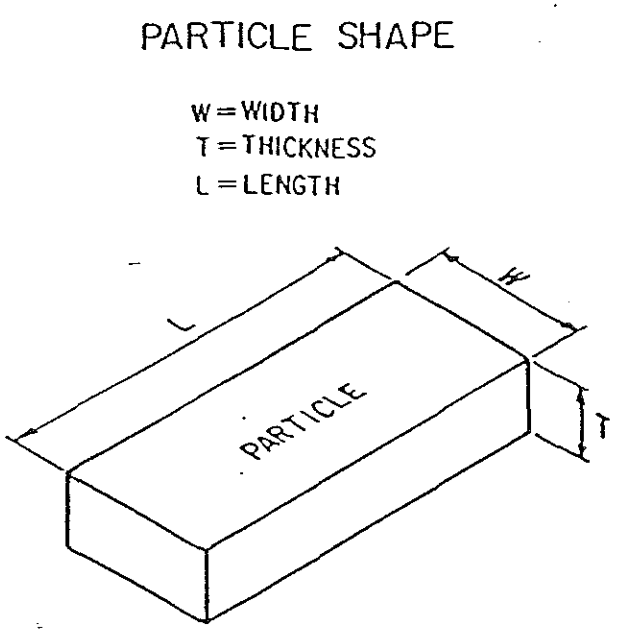



FIG. 4 Criteria for Particle Shape

TABLE 3 Criteria for Describing Moisture Condition

| Description | Criteria |
|---|---|
| Dry | Absence of moisture, dusty, dry to the touch |
| Moist | Damp but no visible water |
| Wet | Visible free water, usually soil is below water table |

locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

TABLE 4 Criteria for Describing the Reaction With HCl

| Description | Criteria |
|---|---|
| None | No visible reaction |
| Weak | Some reaction, with bubbles forming slowly |
| Strong | Violent reaction, with bubbles forming immediately |

TABLE 5 Criteria for Describing Consistency

| Description | Criteria |
|---|---|
| Very soft | Thumb will penetrate soil more than 1 in. (25 mm) |
| Soft | Thumb will penetrate soil about 1 in. (25 mm) |
| Firm | Thumb will indent soil about ¼ in. (6 mm) |
| Hard | Thumb will not indent soil but readily indented with thumbnail |
| Very hard | Thumbnail will not indent soil |

10.7 *Consistency*—For intact fine-grained soil, describe the consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpre-

TABLE 6 Criteria for Describing Cementation

| Description | Criteria |
|---|---|
| Weak | Crumbles or breaks with handling or little finger pressure |
| Moderate | Crumbles or breaks with considerable finger pressure |
| Strong | Will not crumble or break with finger pressure |

TABLE 7 Criteria for Describing Structure

| Description | Criteria |
|---|---|
| .atified | Alternating layers of varying material or color with layers at least 6 mm thick; note thickness |
| aminated | Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness |
| ssured | Breaks along definite planes of fracture with little resistance to fracturing |
| ickensided | Fracture planes appear polished or glossy, sometimes striated |
| ocky | Cohesive soil that can be broken down into small angular lumps which resist further breakdown |
| nsed | Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness |
| omogeneous | Same color and appearance throughout |

ation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in ccordance with other classification systems may be added if dentified as such.

## 1. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in arious stages of decomposition that has a fibrous to morphous texture, usually a dark brown to black color, and n organic odor, shall be designated as a highly organic soil nd shall be identified as peat, PT, and not subjected to the dentification procedures described hereafter.

## 2. Preparation for Identification

12.1 The soil identification portion of this practice is ased on the portion of the soil sample that will pass a 3-in. 75-mm) sieve. The larger than 3-in. (75-mm) particles must e removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 8—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 9—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term ce, for example, trace of fines. A trace is not to be nsidered in the total of 100 % for the components.

## 3. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

## 4. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength:*

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 10—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy:*

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on

TABLE 8 Criteria for Describing Dry Strength

| Description | Criteria |
|---|---|
| None | The dry specimen crumbles into powder with mere pressure of handling |
| Low | The dry specimen crumbles into powder with some finger pressure |
| Medium | The dry specimen breaks into pieces or crumbles with considerable finger pressure |
| High | The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface |
| Very high | The dry specimen cannot be broken between the thumb and a hard surface |

TABLE 9 Criteria for Describing Dilatancy

| Description | Criteria |
|---|---|
| None | No visible change in the specimen |
| Slow | Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing |
| Rapid | Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing |

TABLE 10 Criteria for Describing Toughness

| Description | Criteria |
|---|---|
| Low | Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft |
| Medium | Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness |
| High | Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness |

the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness:*

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils:*

TABLE 11 Criteria for Describing Plasticity

| Description | Criteria |
|---|---|
| Nonplastic | A ⅛-in. (3-mm) thread cannot be rolled at any water content |
| Low | The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit |
| Medium | The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit |
| High | It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit |

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 11—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils:*

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 12—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words "with sand" or "with gravel" (whichever is more predominant) shall be added to the group name. For example: "lean clay with sand, CL" or "silt with gravel, ML" (see Figs. 1a and 1b). If the percentage of sand is equal to the percentage of gravel, use "with sand."

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if there appears to be more sand than gravel. Add the word "gravelly" if there appears to be more gravel than sand. For example: "sandy lean clay, CL", "gravelly fat clay, CH", or "sandy silt, ML" (see Figs. 1a and 1b). If the percentage of sand is equal to the percent of gravel, use "sandy."

## 15. Procedure for Identifying Coarse-Grained Soils (Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

| Soil Symbol | Dry Strength | Dilatancy | Toughness |
|---|---|---|---|
| ML | None to low | Slow to rapid | Low or thread cannot be formed |
| CL | Medium to high | None to slow | Medium |
| MH | Low to medium | None to slow | Low to medium |
| CH | High to very high | None | High |

15.2 The soil is a *sand* if the percentage of gravel is timated to be equal to or less than the percentage of sand.

5.3 The soil is a *clean gravel* or *clean sand* if the .centage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as *well-graded sand*, SW, if it has a wide range of particle sizes nd substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as *poorly graded sand*, SP, if it consists predominantly of one ize (uniformly graded), or it has a wide range of sizes with ome intermediate sizes obviously missing (gap or skip raded).

15.4 The soil is either a *gravel with fines* or a *sand with nes* if the percentage of fines is estimated to be 15 % or ıore.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey and*, SC, if the fines are clayey as determined by the rocedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty and*, SM, if the fines are silty as determined by the rocedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the oil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean ravel or sand (GW, GP, SW, SP) and the second symbol hall correspond to a gravel or sand with fines (GC, GM, SC, M).

15.5.2 The group name shall correspond to the first group mbol plus the words "with clay" or "with silt" to indicate plasticity characteristics of the fines. For example: -graded gravel with clay, GW-GC" or "poorly graded with silt, SP-SM" (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but ontains an estimated 15 % or more of the other coarse-rained constituent, the words "with gravel" or "with sand" hall be added to the group name. For example: "poorly raded gravel with sand, GP" or "clayey sand with gravel, C" (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, both, the words "with cobbles" or "with cobbles and oulders" shall be added to the group name. For example: ilty gravel with cobbles, GM."

## 16. Report

16.1 The report shall include the information as to origin, nd the items indicated in Table 13.

NOTE 13—*Example: Clayey Gravel with Sand and Cobbles*. GC—bout 50 % fine to coarse, subrounded to subangular gravel; about 30 % e to coarse, subrounded sand; about 20 % fines with medium sticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

*For intact samples:*

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

NOTE 14—Other examples of soil descriptions and identification are given in Appendixes X1 and X2.

NOTE 15—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

*Trace*—Particles are present but estimated to be less than 5 %

*Few*—5 to 10 %

*Little*—15 to 25 %

*Some*—30 to 45 %

*Mostly*—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

## 17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

## 18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

## APPENDIXES

(Nonmandatory Information)

## X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

*In-Place Conditions*—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft$^3$; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

## X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incororated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as "Sandy Lean Clay (CL)"; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; "Poorly Graded Sand with Silt (SP-SM)"; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

## X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the

percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

## X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

Designation: D 2487 – 90



# Standard Test Method for Classification of Soils for Engineering Purposes[1]

This standard is issued under the fixed designation D 2487; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This test method has been approved for use by agencies of the Department of Defense. Consult the DOD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.*

## 1. Scope

1.1 This test method describes a system for classifying mineral and organo-mineral soils for engineering purposes based on laboratory determination of particle-size characteristics, liquid limit, and plasticity index and shall be used when precise classification is required.

NOTE 1—Use of this standard will result in a single classification group symbol and group name except when a soil contains 5 to 12 % fines or when the plot of the liquid limit and plasticity index values falls into the crosshatched area of the plasticity chart. In these two cases, a dual symbol is used, for example, GP-GM, CL-ML. When the laboratory test results indicate that the soil is close to another soil classification group, the borderline condition can be indicated with two symbols separated by a slash. The first symbol should be the one based on this standard, for example, CL/CH, GM/SM, SC/CL. Borderline symbols are particularly useful when the liquid limit value of clayey soils is close to 50. These soils can have expansive characteristics and the use of a borderline symbol (CL/CH, CH/CL) will alert the user of the assigned classifications of expansive potential.

1.2 The group symbol portion of this sytem is based on laboratory tests performed on the portion of a soil sample passing the 3-in. (75-mm) sieve (see Specification E 11).

1.3 As a classification system, this test method is limited to naturally occurring soils.

NOTE 2—The group names and symbols used in this test method may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. See Appendix X2.

1.4 This test method is for qualitative application only.

NOTE 3—When quantitative information is required for detailed designs of important structures, this test method must be supplemented by laboratory tests or other quantitative data to determine performance characteristics under expected field conditions.

1.5 The system is based on the widely recognized Unified Soil Classification System which was adopted by several U.S. Government agencies in 1952 as an outgrowth of the Airfield Classification System developed by A. Casagrande.[2]

1.6 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

C 117 Test Method for Materials Finer Than 75-μm (No. 200) Sieve in Mineral Aggregates by Washing[3]

C 136 Method for Sieve Analysis of Fine and Coarse Aggregates[3]

C 702 Practice for Reducing Field Samples of Aggregate to Testing Size[3]

D 420 Practice for Investigating and Sampling Soil and Rock for Engineering Purposes[4]

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[4]

D 422 Method for Particle-Size Analysis of Soils[4]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[4]

D 1140 Test Method for Amount of Material in Soils Finer than the No. 200 (75-μm) Sieve[4]

D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures[4]

D 2217 Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[4]

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[4]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[4]

D 4318 Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils[4]

D 4427 Classification of Peat Samples by Laboratory Testing[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

## 3. Terminology

3.1 *Definitions*—Except as listed below, all definitions are in accordance with Terms and Symbols D 653.

NOTE 4—For particles retained on a 3-in. (75-mm) U.S. standard sieve, the following definitions are suggested:

*Cobbles*—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) U.S. standard sieve, and

*Boulders*—particles of rock that will not pass a 12in. (300-mm) square opening

3.1.1 *gravel*—particles of rock that will pass a 3-in.

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[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

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[2] Casagrande, A., "Classification and Identification of Soils," *Transactions*, ASCE, 1948, p. 901.

[3] *Annual Book of ASTM Standards*, Vol 04.02.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

75-mm) sieve and be retained on a No. 4 (4.75-mm) U.S. andard sieve with the following subdivisions:

*Coarse*—passes 3-in. (75-mm) sieve and retained on ¾-in. (19-mm) sieve, and

*Fine*—passes ¾-in. (19-mm) sieve and retained on No. 4 (4.75-mm) sieve.

3.1.2 *sand*—particles of rock that will pass a No. 4 .75-mm) sieve and be retained on a No. 200 (75-μm) U.S. andard sieve with the following subdivisions:

*Coarse*—passes No. 4 (4.75-mm) sieve and retained on No. 10 (2.00-mm) sieve,

*Medium*—passes No. 10 (2.00-mm) sieve and retained on No. 40 (425-μm) sieve, and

*Fine*—passes No. 40 (425-μm) sieve and retained on No. 200 (75-μm) sieve.

3.1.3 *clay*—soil passing a No. 200 (75-μm) U.S. standard ve that can be made to exhibit plasticity (putty-like properties) within a range of water contents and that exhibits nsiderable strength when air dry. For classification, a clay a fine-grained soil, or the fine-grained portion of a soil, th a plasticity index equal to or greater than 4, and the plot plasticity index versus liquid limit falls on or above the " line.

3.1.4 *silt*—soil passing a No. 200 (75-μm) U.S. standard ve that is nonplastic or very slightly plastic and that hibits little or no strength when air dry. For classification, silt is a fine-grained soil, or the fine-grained portion of a il, with a plasticity index less than 4 or if the plot of asticity index versus liquid limit falls below the "A" line.

3.1.5 *organic clay*—a clay with sufficient organic content nfluence the soil properties. For classification, an organic ay is a soil that would be classified as a clay except that its uid limit value after oven drying is less than 75 % of its uid limit value before oven drying.

3.1.6 *organic silt*—a silt with sufficient organic content to luence the soil properties. For classification, an organic silt a soil that would be classified as a silt except that its liquid mit value after oven drying is less than 75 % of its liquid mit value before oven drying.

3.1.7 *peat*—a soil composed of vegetable tissue in various ges of decomposition usually with an organic odor, a rk-brown to black color, a spongy consistency, and a ture ranging from fibrous to amorphous.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *coefficient of curvature*, Cc—the ratio $(D_{30})^2/(D_{10} \times D_{60})$, where $D_{60}$, $D_{30}$, and $D_{10}$ are the particle diameters corresponding to 60, 30, and 10 % finer on the cumulative particle-size distribution curve, respectively.

3.2.2 *coefficient of uniformity*, Cu—the ratio $D_{60}/D_{10}$, where $D_{60}$ and $D_{10}$ are the particle diameters corresponding to 60 and 10 % finer on the cumulative particle-size distribution curve, respectively.

## Summary of Test Method

4.1 As illustrated in Table 1, this classification system identifies three major soil divisions: coarse-grained soils, fine-grained soils, and highly organic soils. These three divisions are further subdivided into a total of 15 basic soil groups.

4.2 Based on the results of visual observations and prescribed laboratory tests, a soil is catalogued according to the 15 soil groups, assigned a group symbol(s) and name, and thereby classified. The flow charts, Fig. 1 for fine-grained soils, and Fig. 2 for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name.

## 5. Significance and Use

5.1 This test method classifies soils from any geographic location into categories representing the results of prescribed laboratory tests to determine the particle-size characteristics, the liquid limit, and the plasticity index.

5.2 The assigning of a group name and symbol(s) along with the descriptive information required in Practice D 2488 can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.3 The various groupings of this classification system have been devised to correlate in a general way with the engineering behavior of soils. This test method provides a useful first step in any field or laboratory investigation for geotechnical engineering purposes.

5.4 This test method may also be used as an aid in training personnel in the use of Practice D 2488.

5.5 This test method may be used in combination with Practice D 4083 when working with frozen soils.

## 6. Apparatus

6.1 In addition to the apparatus that may be required for obtaining and preparing the samples and conducting the prescribed laboratory tests, a plasticity chart, similar to Fig. 3, and a cumulative particle-size distribution curve, similar to Fig. 4, are required.

NOTE 5—The "U" line shown on Fig. 3 has been empirically determined to be the approximate "upper limit" for natural soils. It is a good check against erroneous data, and any test results that plot above or to the left of it should be verified.

## 7. Sampling

7.1 Samples shall be obtained and identified in accordance with a method or methods, recommended in Recommended Practice D 420 or by other accepted procedures.

7.2 For accurate identification, the minimum amount of test sample required for this test method will depend on which of the laboratory tests need to be performed. Where only the particle-size analysis of the sample is required, specimens having the following minimum dry weights are required:

| Maximum Particle Size, Sieve Opening | Minimum Specimen Size, Dry Weight |
|---|---|
| 4.75 mm (No. 4) | 100 g (0.25 lb) |
| 9.5 mm (⅜ in.) | 200 g (0.5 lb) |
| 19.0 mm (¾ in.) | 1.0 kg (2.2 lb) |
| 38.1 mm (1½ in.) | 8.0 kg (18 lb) |
| 75.0 mm (3 in.) | 60.0 kg (132 lb) |

Whenever possible, the field samples should have weights two to four times larger than shown.

7.3 When the liquid and plastic limit tests must also be performed, additional material will be required sufficient to provide 150 g to 200 g of soil finer than the No. 40 (425-μm) sieve.

7.4 If the field sample or test specimen is smaller than the minimum recommended amount, the report shall include an appropriate remark.

TABLE 1 Soil Classification Chart

| Criteria for Assigning Group Symbols and Group Names Using Laboratory Tests[A] | | | | Soil Classification | |
|---|---|---|---|---|---|
| | | | | Group Symbol | Group Name[B] |
| Coarse-Grained Soils<br>More than 50 % retained on No. 200 sieve | Gravels<br>More than 50 % of coarse fraction retained on No. 4 sieve | Clean Gravels<br>Less than 5 % fines[C] | $Cu \geq 4$ and $1 \leq Cc \leq 3$[E] | GW | Well-graded gravel[F] |
| | | | $Cu < 4$ and/or $1 > Cc > 3$[E] | GP | Poorly graded gravel[F] |
| | | Gravels with Fines More than 12 % fines[C] | Fines classify as ML or MH | GM | Silty gravel[F,G,H] |
| | | | Fines classify as CL or CH | GC | Clayey gravel[F,G,H] |
| | Sands<br>50 % or more of coarse fraction passes No. 4 sieve | Clean Sands<br>Less than 5 % fines[D] | $Cu \geq 6$ and $1 \leq Cc \leq 3$[E] | SW | Well-graded sand |
| | | | $Cu < 6$ and/or $1 > Cc > 3$[E] | SP | Poorly graded sand[I] |
| | | Sands with Fines<br>More than 12 % fines[D] | Fines classify as ML or MH | SM | Silty sand[G,H,I] |
| | | | Fines classify as CL or CH | SC | Clayey sand[G,H,I] |
| Fine-Grained Soils<br>50 % or more passes the No. 200 sieve | Silts and Clays<br>Liquid limit less than 50 | Inorganic | PI > 7 and plots on or above "A" line[J] | CL | Lean clay[K,L,M] |
| | | | PI < 4 or plots below "A" line[J] | ML | Silt[K,L,M] |
| | | organic | $\frac{\text{Liquid limit - oven dried}}{\text{Liquid limit - not dried}} < 0.75$ | OL | Organic clay[K,L,M,N]<br>Organic silt[K,L,M,O] |
| | Silts and Clays<br>Liquid limit 50 or more | Inorganic | PI plots on or above "A" line | CH | Fat clay[K,L,M] |
| | | | PI plots below "A" line | MH | Elastic silt[K,L,M] |
| | | organic | $\frac{\text{Liquid limit - oven dried}}{\text{Liquid limit - not dried}} < 0.75$ | OH | Organic clay[K,L,M,P]<br>Organic silt[K,L,M,Q] |
| Highly organic soils | | Primarily organic matter, dark in color, and organic odor | | PT | Peat |

[A] Based on the material passing the 3-in. (75-mm) sieve.

[B] If field sample contained cobbles or boulders, or both, add "with cobbles or boulders, or both" to group name.

[C] Gravels with 5 to 12 % fines require dual symbols:
GW-GM well-graded gravel with silt
GW-GC well-graded gravel with clay
GP-GM poorly graded gravel with silt
GP-GC poorly graded gravel with clay

[D] Sands with 5 to 12 % fines require dual symbols:
SW-SM well-graded sand with silt
SW-SC well-graded sand with clay
SP-SM poorly graded sand with silt
SP-SC poorly graded sand with clay

[E] $Cu = D_{60}/D_{10}$ $\quad Cc = \frac{(D_{30})^2}{D_{10} \times D_{60}}$

[F] If soil contains ≥ 15 % sand, add "with sand" to group name.

[G] If fines classify as CL-ML, use dual symbol GC-GM, or SC-SM.

[H] If fines are organic, add "with organic fines" to group name.

[I] If soil contains ≥ 15 % gravel, add "with gravel" to group name.

[J] If Atterberg limits plot in hatched area, soil is a CL-ML, silty clay.

[K] If soil contains 15 to 29 % plus No. 200, add "with sand" or "with gravel," whichever is predominant.

[L] If soil contains ≥ 30 % plus No. 200, predominantly sand, add "sandy" to group name.

[M] If soil contains ≥ 30 % plus No. 200, predominantly gravel, add "gravelly" to group name.

[N] PI ≥ 4 and plots on or above "A" line.

[O] PI < 4 or plots below "A" line.

[P] PI plots on or above "A" line.

[Q] PI plots below "A" line.

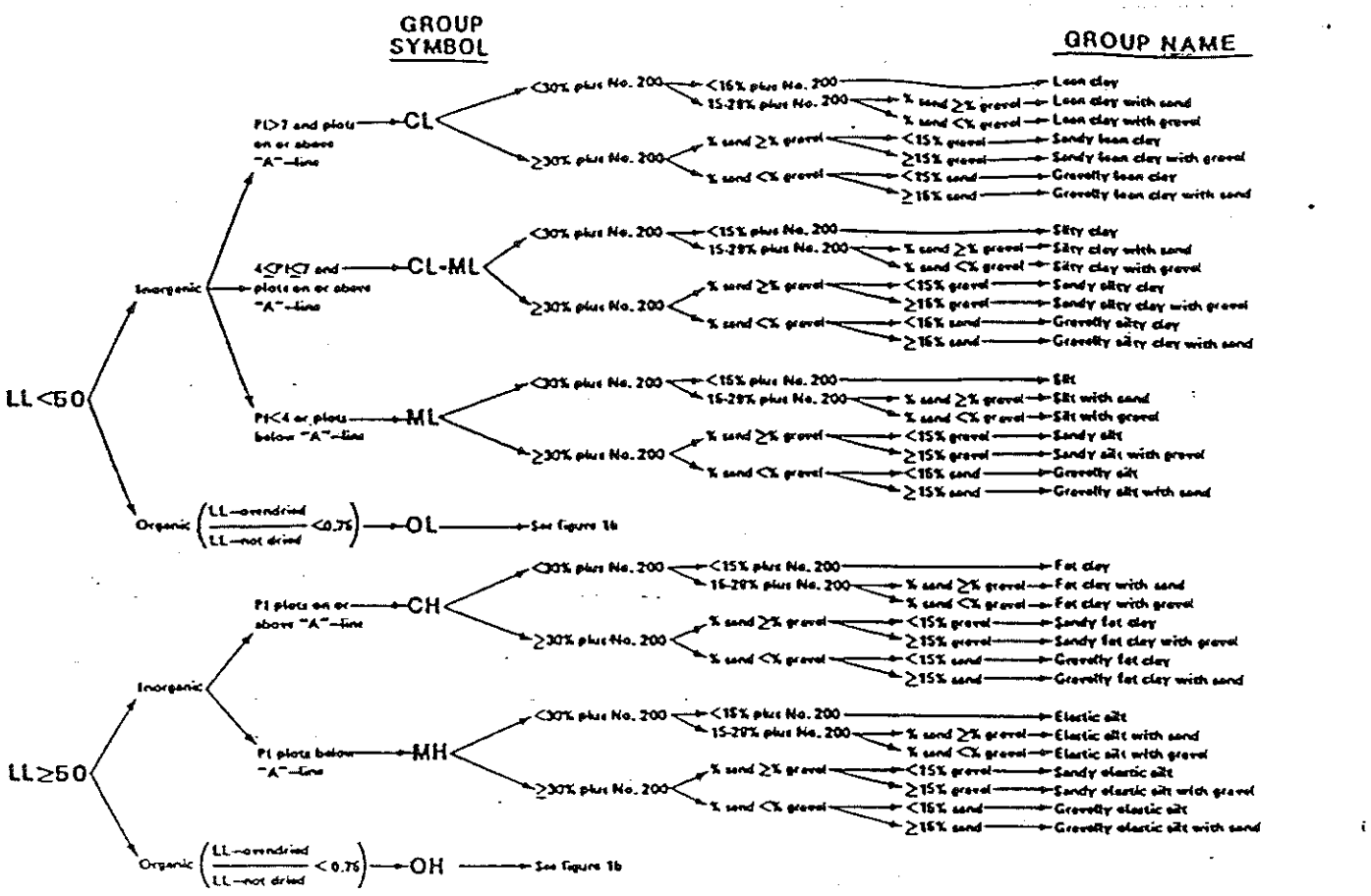


FIG. 1a Flow Chart for Classifying Fine-Grained Soil (50 % or More Passes No. 200 Sieve)

8. Classification of Peat

8.1 A sample composed primarily of vegetable tissue in various stages of decomposition and has a fibrous to amorphous texture, a dark-brown to black color, and an organic odor should be designated as a highly organic soil and shall be classified as peat, PT, and not subjected to the classification procedures described hereafter.

8.2 If desired, classification of type of peat can be performed in accordance with Classification D 4427.

9. Preparation for Classification

9.1 Before a soil can be classified according to this test method, generally the particle-size distribution of the minus 3-in. (75-mm) material and the plasticity characteristics of the minus No. 40 (425-μm) sieve material must be determined. See 9.8 for the specific required tests.

9.2 The preparation of the soil specimen(s) and the testing for particle-size distribution and liquid limit and plasticity index shall be in accordance with accepted standard procedures. Two procedures for preparation of the soil specimens for testing for soil classification purposes are given in Appendixes X3 and X4. Appendix X3 describes the wet preparation method and is the preferred method for cohesive soils that have never dried out and for organic soils.

9.3 When reporting soil classifications determined by this test method, the preparation and test procedures used shall be reported or referenced.

9.4 Although the test procedure used in determining the particle-size distribution or other considerations may require a hydrometer analysis of the material, a hydrometer analysis is not necessary for soil classification.

9.5 The percentage (by dry weight) of any plus 3-in. (75-mm) material must be determined and reported as auxiliary information.

9.6 The maximum particle size shall be determined (measured or estimated) and reported as auxiliary information.

9.7 When the cumulative particle-size distribution is required, a set of sieves shall be used which include the following sizes (with the largest size commensurate with the maximum particle size) with other sieve sizes as needed or required to define the particle-size distribution:

3-in. (75-mm)
¾-in.(19.0-mm)
No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 40 (425-μm)
No. 200 (75-μm)

9.8 The tests required to be performed in preparation for classification are as follows:

9.8.1 For soils estimated to contain less than 5 % fines, a plot of the cumulative particle-size distribution curve of the fraction coarser than the No. 200 (75-μm) sieve is required. The cumulative particle-size distribution curve may be plotted on a graph similar to that shown in Fig. 4.

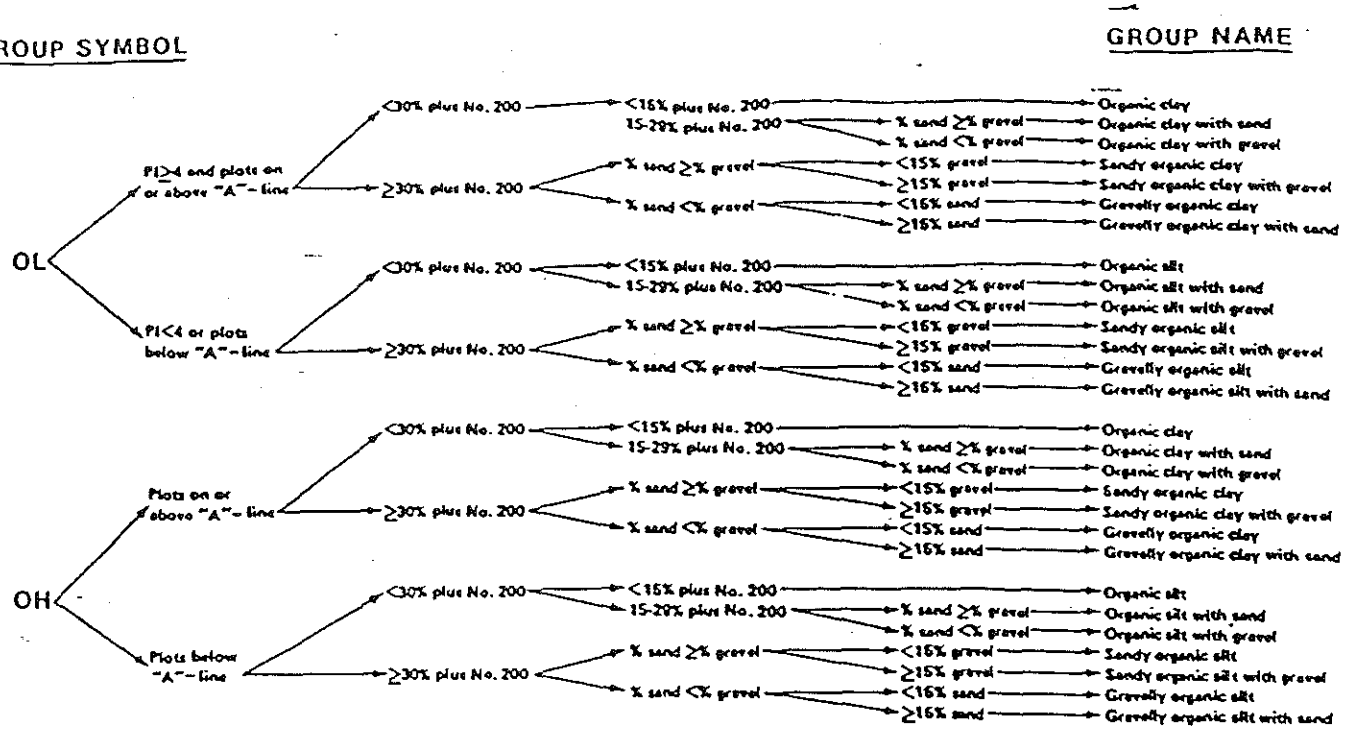


FIG. 1b Flow Chart for Classifying Organic Fine-Grained Soil (50 % or More Passes No. 200 Sieve)

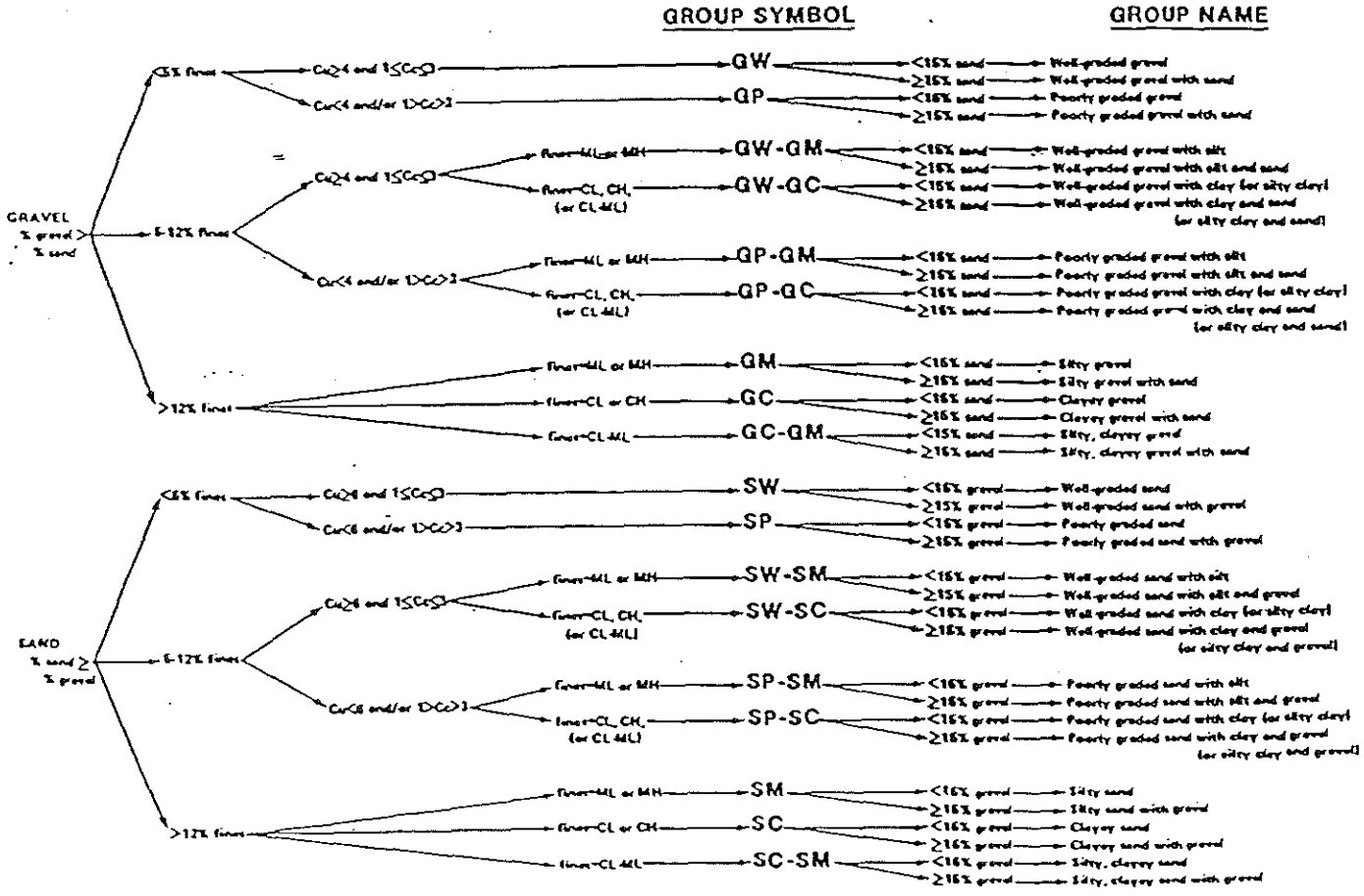


FIG. 2 Flow Chart for Classifying Coarse-Grained Soils (More Than 50 % Retained on No. 200 Sieve)

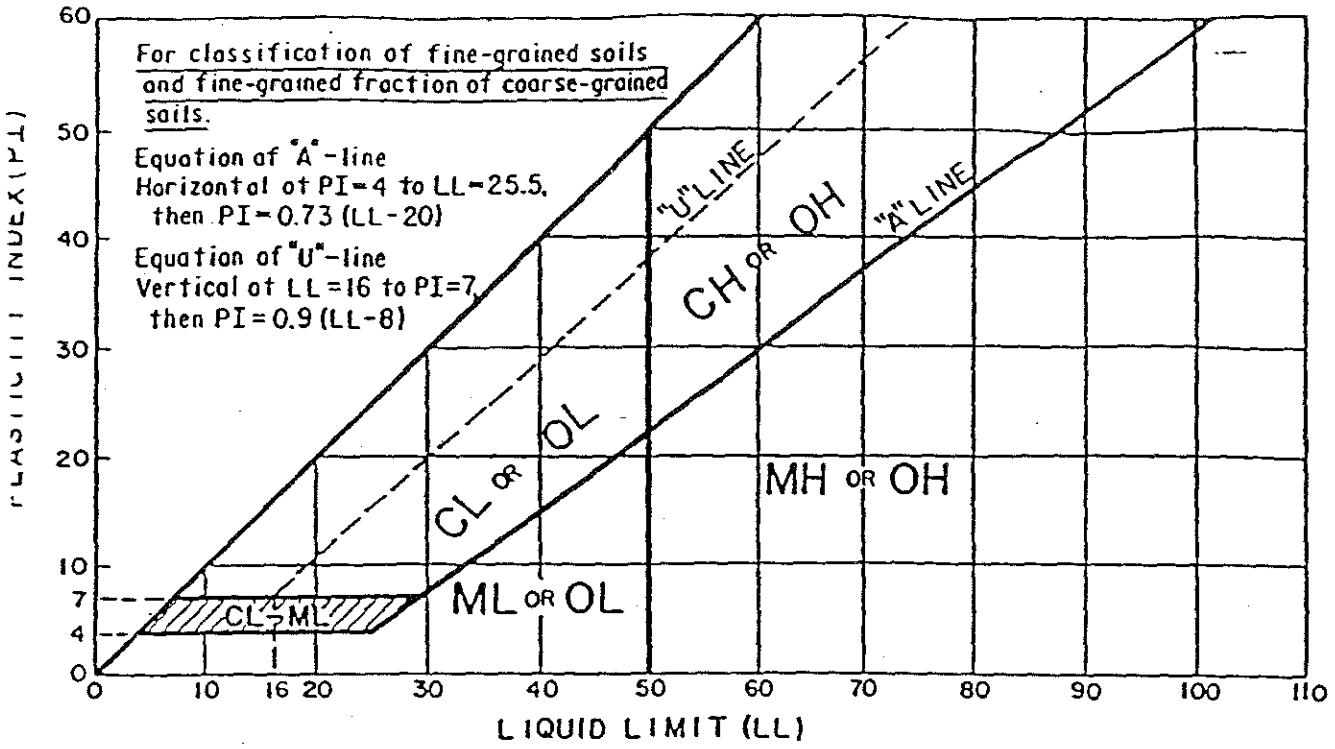


FIG. 3 Plasticity Chart

.2 For soils estimated to contain 5 to 15 % fines, a ulative particle-size distribution curve, as described in .1, is required, and the liquid limit and plasticity index are uired.

.8.2.1 If sufficient material is not available to determine liquid limit and plasticity index, the fines should be mated to be either silty or clayey using the procedures cribed in Practice D 2488 and so noted in the report.

.8.3 For soils estimated to contain 15 % or more fines, a rmination of the percent fines, percent sand, and percent vel is required, and the liquid limit and plasticity index required. For soils estimated to contain 90 % fines or re, the percent fines, percent sand, and percent gravel be estimated using the procedures described in Practice 488 and so noted in the report.

Preliminary Classification Procedure

).1 Class the soil as fine-grained if 50 % or more by dry ght of the test specimen passes the No. 200 (75-μm) sieve follow Section 11.

).2 Class the soil as coarse-grained if more than 50 % by weight of the test specimen is retained on the No. 200 μm) sieve and follow Section 12.

Procedure for Classification of Fine-Grained Soils (50 % or more by dry weight passing the No. 200 (75-μm) sieve)

The soil is an inorganic clay if the position of the ity index versus liquid limit plot, Fig. 3, falls on or e the "A" line, the plasticity index is greater than 4, and resence of organic matter does not influence the liquid as determined in 11.3.2.

NOTE 6—The plasticity index and liquid limit are determined on the minus No. 40 (425 μm) sieve material.

11.1.1 Classify the soil as a *lean clay*, CL, if the liquid limit is less than 50. See area identified as CL on Fig. 3.

11.1.2 Classify the soil as a *fat clay*, CH, if the liquid limit is 50 or greater. See area identified as CH on Fig. 3.

NOTE 7—In cases where the liquid limit exceeds 110 or the plasticity index exceeds 60, the plasticity chart may be expanded by maintaining the same scale on both axes and extending the "A" line at the indicated slope.

11.1.3 Classify the soil as a *silty clay*, CL-ML, if the position of the plasticity index versus liquid limit plot falls on or above the "A" line and the plasticity index is in the range of 4 to 7. See area identified as CL-ML on Fig. 3.

11.2 The soil is an inorganic silt if the position of the plasticity index versus liquid limit plot, Fig. 3, falls below the "A" line or the plasticity index is less than 4, and presence of organic matter does not influence the liquid limit as determined in 11.3.2.

11.2.1 Classify the soil as a *silt*, ML, if the liquid limit is less than 50. See area identified as ML on Fig. 3.

11.2.2 Classify the soil as an *elastic silt*, MH, if the liquid limit is 50 or greater. See area identified as MH on Fig. 3.

11.3 The soil is an organic silt or clay if organic matter is present in sufficient amounts to influence the liquid limit as determined in 11.3.2.

11.3.1 If the soil has a dark color and an organic odor when moist and warm, a second liquid limit test shall be performed on a test specimen which has been oven dried at 110 ± 5°C to a constant weight, typically over night.

11.3.2 The soil is an organic silt or organic clay if the liquid limit after oven drying is less than 75 % of the liquid

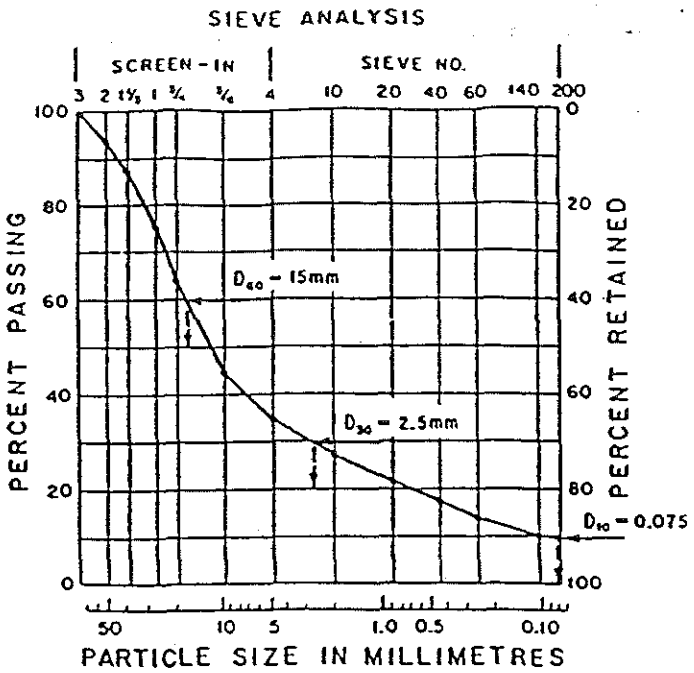


FIG. 4 Cumulative Particle-Size Plot

limit of the original specimen determined before oven drying (see Procedure B of Practice D 2217).

11.3.3 Classify the soil as an *organic silt* or *organic clay*, OL, if the liquid limit (not oven dried) is less than 50 %. Classify the soil as an *organic silt*, OL, if the plasticity index is less than 4, or the position of the plasticity index versus liquid limit plot falls below the "A" line. Classify the soil as an *organic clay*, OL, if the plasticity index is 4 or greater and the position of the plasticity index versus liquid limit plot falls on or above the "A" line. See area identified as OL (or CL-ML) on Fig. 3.

11.3.4 Classify the soil as an *organic clay* or *organic silt*, OH, if the liquid limit (not oven dried) is 50 or greater. Classify the soil as an *organic silt*, OH, if the position of the plasticity index versus liquid limit plot falls below the "A" line. Classify the soil as an *organic clay*, OH, if the position of the plasticity index versus liquid-limit plot falls on or above the "A" line. See area identified as OH on Fig. 3.

11.4 If less than 30 % but 15 % or more of the test specimen is retained on the No. 200 (75-μm) sieve, the words "with sand" or "with gravel" (whichever is predominant) shall be added to the group name. For example, lean clay with sand, CL; silt with gravel, ML. If the percent of sand is equal to the percent of gravel, use "with sand."

11.5 If 30 % or more of the test specimen is retained on the No. 200 (75-μm) sieve, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if 30 % or more of the test specimen is retained on the No. 200 (75-μm) sieve and the coarse-grained portion is predominantly sand. Add the word "gravelly" if 30 % or more of the test specimen is retained on the No. 200 (75-μm) sieve and the coarse-grained portion is predominantly gravel. For example, sandy lean clay, CL; gravelly fat clay, CH; sandy silt, ML. If the percent of sand is equal to the percent of gravel, use "sandy."

## 12. Procedure for Classification of Coarse-Grained Soils (more than 50 % retained on the No. 200 (75-μm) sieve)

12.1 Class the soil as gravel if more than 50 % of the coarse fraction [plus No. 200 (75-μm) sieve] is retained on the No. 4 (4.75-mm) sieve.

12.2 Class the soil as sand if 50 % or more of the coarse fraction [plus No. 200 (75-μm) sieve] passes the No. 4 (4.75-mm) sieve.

12.3 If 12 % or less of the test specimen passes the No. 200 (75-μm) sieve, plot the cumulative particle-size distribution, Fig. 4, and compute the coefficient of uniformity, Cu, and coefficient of curvature, Cc, as given in Eqs 1 and 2.

$$Cu = D_{60}/D_{10} \quad (1)$$

$$Cc = (D_{30})^2/(D_{10} \times D_{60}) \quad (2)$$

where:

$D_{10}$, $D_{30}$, and $D_{60}$ = the particle-size diameters corresponding to 10, 30, and 60 %, respectively, passing on the cumulative particle-size distribution curve, Fig. 4.

NOTE 8—It may be necessary to extrapolate the curve to obtain the $D_{10}$ diameter.

12.3.1 If less than 5 % of the test specimen passes the No. 200 (75-μm) sieve, classify the soil as a *well-graded gravel*, GW, or *well-graded sand*, SW, if Cu is greater than 4.0 for gravel or greater than 6.0 for sand, and Cc is at least 1.0 but not more than 3.0.

12.3.2 If less than 5 % of the test specimen passes the No. 200 (75-μm) sieve, classify the soil as *poorly graded gravel*,

GP, or *poorly graded sand*, SP, if either the Cu or the Cc iteria for well-graded soils are not satisfied.

12.4 If more than 12 % of the test specimen passes the No. 200 (75-μm) sieve, the soil shall be considered a coarse-grained soil with fines. The fines are determined to be either clayey or silty based on the plasticity index versus liquid limit plot on Fig. 3. (See 9.8.2.1 if insufficient material available for testing). (See NOTE 6)

12.4.1 Classify the soil as a *clayey gravel*, GC, or *clayey sand*, SC, if the fines are clayey, that is, the position of the plasticity index versus liquid limit plot, Fig. 3, falls on or above the "A" line and the plasticity index is greater than 7.

12.4.2 Classify the soil as a *silty gravel*, GM, or *silty sand*, SM, if the fines are silty, that is, the position of the plasticity index versus liquid limit plot, Fig. 3, falls below the "A" line or the plasticity index is less than 4.

12.4.3 If the fines plot as a silty clay, CL-ML, classify the soil as a *silty, clayey gravel*, GC-GM, if it is a gravel or a *silty, clayey sand*, SC-SM, if it is a sand.

12.5 If 5 to 12 % of the test specimen passes the No. 200 (75-μm) sieve, give the soil a dual classification using two group symbols.

12.5.1 The first group symbol shall correspond to that for gravel or sand having less than 5 % fines (GW, GP, SW, SP), and the second symbol shall correspond to a gravel or sand having more than 12 % fines (GC, GM, SC, SM).

12.5.2 The group name shall correspond to the first group symbol plus "with clay" or "with silt" to indicate the plasticity characteristics of the fines. For example, well-graded gravel with clay, GW-GC; poorly graded sand with silt, SP-SM (See 9.8.2.1 if insufficient material available for testing).

NOTE 9—If the fines plot as a *silty clay*, CL-ML, the second group symbol should be either GC or SC. For example, a poorly graded sand with 10 % fines, a liquid limit of 20, and a plasticity index of 6 would be classified as a poorly graded sand with silty clay, SP-SC.

12.6 If the specimen is predominantly sand or gravel but contains 15 % or more of the other coarse-grained constituent, the words "with gravel" or "with sand" shall be added to the group name. For example, poorly graded gravel with sand, clayey sand with gravel.

12.7 If the field sample contained any cobbles or boulders or both, the words "with cobbles," or "with cobbles and boulders" shall be added to the group name. For example, silty gravel with cobbles, GM.

## 13. Report

13.1 The report should include the group name, group symbol, and the results of the laboratory tests. The particle-size distribution shall be given in terms of percent of gravel, sand, and fines. The plot of the cumulative particle-size distribution curve shall be reported if used in classifying the soil. Report appropriate descriptive information according to the procedures in Practice D 2488. A local or commercial name or geologic interpretation for the material may be added at the end of the descriptive information if identified as such. The test procedures used shall be referenced.

NOTE 10—*Example: Clayey Gravel with Sand and Cobbles* (GC)—46 % fine to coarse, hard, subrounded gravel; 30 % fine to coarse, hard, subrounded sand; 24 % clayey fines, LL = 38, PI = 19; weak reaction with HCl; original field sample had 4 % hard, subrounded cobbles; maximum dimension 150 mm.

In-Place Conditions—firm, homogeneous, dry, brown.

Geologic Interpretation—alluvial fan.

NOTE 11—Other examples of soil descriptions are given in Appendix X1.

## 14. Precision and Bias

14.1 This test method provides qualitative data only; therefore, a precision and bias statement is nonapplicable.

## 15. Keywords

15.1 Atterberg limits; classification; clay; gradation; gravel; laboratory classification; organic soils; sand; silt; soil classification; soil tests

# APPENDIXES

(Nonmandatory Information)

## X1. EXAMPLES OF DESCRIPTIONS USING SOIL CLASSIFICATION

X1.1 The following examples show how the information required in 13.1 can be reported. The appropriate descriptive information from Practice D 2488 is included for illustrative purposes. The additional descriptive terms that would accompany the soil classification should be based on the intended use of the classification and the individual circumstances.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—73 % fine to coarse, hard, subangular gravel; 23 % fine to coarse, hard, subangular sand; 4 % fines; Cc = 2.7, Cu = 12.4.

X1.1.2 *Silty Sand with Gravel (SM)*—61 % predominantly fine sand; 23 % silty fines, LL = 33, PI = 6; 16 % fine, hard, subrounded gravel; no reaction with HCl; (field sample smaller than recommended). *In-Place Conditions*—Firm, stratified and contains lenses of silt 1 to 2 in. thick, moist, brown to gray; in-place density = 106 lb/ft$^3$ and in-place moisture = 9 %.

X1.1.3 *Organic Clay (OL)*—100 % fines, LL (not dried) = 32, LL (oven dried) = 21, PI (not dried) = 10; wet, dark brown, organic odor, weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—74 % fine to coarse, hard, subangular reddish sand; 26 % organic and silty dark-brown fines, LL (not dried) = 37, LL (oven dried) = 26, PI (not dried) = 6, wet, weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—78 % fine to coarse, hard, subrounded to subangular gravel; 16 % fine to coarse, hard, subrounded

to subangular sand; 6 % silty (estimated) fines; moist, brown; no reaction with HCl; original field sample had 7 % hard, subrounded cobbles and 2 % hard, subrounded boulders with a maximum dimension of 18 in.

## X2. USING SOIL CLASSIFICATION AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, ETC.

X2.1 The group names and symbols used in this test method may be used as a descriptive system applied to materials that exist in situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, etc.).

X2.2 Materials such as shells, crushed rock, slag, etc., should be identified as such. However, the procedures used in this method for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, a classification in accordance with this test method may be assigned to aid in describing the material.

X2.3 If a classification is used, the group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how soil classifications could be incorporated into a description system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. pieces of shale from power auger hole, dry, brown, no reaction with HCl. After laboratory processing by slaking in water for 24 h, material classified as "Sandy Lean Clay (CL)"—61 % clayey fines, LL = 37, PI = 16; 33 % fine to medium sand; 6 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; "Poorly Graded Sand with Silt (SP-SM)"—91 % fine to medium sand; 9 % silty (estimated) fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—62 % gravel-size broken shells; 31 % sand and sand-size shell pieces; 7 % fines; would be classified as "Poorly Graded Gravel with Sand (GP)".

X2.4.4 *Crushed Rock*—Processed gravel and cobbles from Pit No. 7; "Poorly Graded Gravel (GP)"—89 % fine, hard, angular gravel-size particles; 11 % coarse, hard, angular sand-size particles, dry, tan; no reaction with HCl; Cc = 2.4, Cu = 0.9.

## X3. PREPARATION AND TESTING FOR CLASSIFICATION PURPOSES BY THE WET METHOD

X3.1 This appendix describes the steps in preparing a soil sample for testing for purposes of soil classification using a wet-preparation procedure.

X3.2 Samples prepared in accordance with this procedure should contain as much of their natural water content as possible and every effort should be made during obtaining, preparing, and transportating the samples to maintain the natural moisture.

X3.3 The procedures to be followed in this test method assume that the field sample contains fines, sand, gravel, and plus 3-in. (75-mm) particles and the cumulative particle-size distribution plus the liquid limit and plasticity index values are required (see 9.8). Some of the following steps may be omitted when they are not applicable to the soil being tested.

X3.4 If the soil contains plus No. 200 (75-μm) particles that would degrade during dry sieving, use a test procedure for determining the particle-size characteristics that prevents this degradation.

X3.5 Since this classification system is limited to the portion of a sample passing the 3-in. (75-mm) sieve, the plus 3-in. (75-mm) material shall be removed prior to the determination of the particle-size characteristics and the liquid limit and plasticity index.

X3.6 The portion of the field sample finer than the 3-in. (75-mm) sieve shall be obtained as follows:

X3.6.1 Separate the field sample into two fractions on a 3-in. (75-mm) sieve, being careful to maintain the natural water content in the minus 3-in. (75-mm) fraction. Any particles adhering to the plus 3-in. (75-mm) particles shall be brushed or wiped off and placed in the fraction passing the 3-in. (75-mm) sieve.

X3.6.2 Determine the air-dry or oven-dry weight of the fraction retained on the 3-in. (75-mm) sieve. Determine the total (wet) weight of the fraction passing the 3-in. (75-mm) sieve.

X3.6.3 Thoroughly mix the fraction passing the 3-in. (75-mm) sieve. Determine the water content, in accordance with Method D 2216, of a representative specimen with a minimum dry weight as required in 7.2. Save the water-content specimen for determination of the particle-size analysis in accordance with X3.8.

X3.6.4 Compute the dry weight of the fraction passing the 3-in. (75-mm) sieve based on the water content and total (wet) weight. Compute the total dry weight of the sample and calculate the percentage of material retained on the 3-in. (75-mm) sieve.

X3.7 Determine the liquid limit and plasticity index as follows:

X3.7.1 If the soil disaggregates readily, mix on a clean, hard surface and select a representative sample by quartering in accordance with Methods C 702.

X3.7.1.1 If the soil contains coarse-grained particles coated with and bound together by tough clayey material, take extreme care in obtaining a representative portion of the No. 40 (425-μm) fraction. Typically, a larger portion than normal has to be selected, such as the minimum weights required in 7.2.

X3.7.1.2 To obtain a representative specimen of a basically cohesive soil, it may be advantageous to pass the soil through a ¾-in. (19-mm) sieve or other convenient size so the material can be more easily mixed and then quartered or split to obtain the representative specimen.

X3.7.2 Process the representative specimen in accordance with Procedure B of Practice D 2217.

X3.7.3 Perform the liquid-limit test in accordance with Test Method D 4318, except the soil shall not be air dried prior to the test.

X3.7.4 Perform the plastic-limit test in accordance with Test Method D 4318, except the soil shall not be air dried prior to the test, and calculate the plasticity index.

X3.8 Determine the particle-size distribution as follows:

X3.8.1 If the water content of the fraction passing the 3-in. (75-mm) sieve was required (X3.6.3), use the water-content specimen for determining the particle-size distribution. Otherwise, select a representative specimen in accordance with Practice C 702 with a minimum dry weight as required in 7.2.

X3.8.2 If the cumulative particle-size distribution including a hydrometer analysis is required, determine the particle-size distribution in accordance with Method D 422. See 9.7 for the set of required sieves.

X3.8.3 If the cumulative particle-size distribution without a hydrometer analysis is required, determine the particle-size distribution in accordance with Method C 136. See 9.7 for the set of required sieves. The specimen should be soaked until all clayey aggregations have softened and then washed in accordance with Test Method C 117 prior to performing the particle-size distribution.

X3.8.4 If the cumulative particle-size distribution is not required, determine the percent fines, percent sand, and percent gravel in the specimen in accordance with Test Method C 117, being sure to soak the specimen long enough to soften all clayey aggregations, followed by Method C 136 using a nest of sieves which shall include a No. 4 (4.75-mm) sieve and a No. 200 (75-μm) sieve.

X3.8.5 Calculate the percent fines, percent sand, and percent gravel in the minus 3-in. (75-mm) fraction for classification purposes.

## X4. AIR-DRIED METHOD OF PREPARATION OF SOILS FOR TESTING FOR CLASSIFICATION PURPOSES

X4.1 This appendix describes the steps in preparing a soil sample for testing for purposes of soil classification when air-drying the soil before testing is specified or desired or when the natural moisture content is near that of an air-dried state.

X4.2 If the soil contains organic matter or mineral colloids that are irreversibly affected by air drying, the wet-preparation method as described in Appendix X3 should be used.

X4.3 Since this classification system is limited to the portion of a sample passing the 3-in. (75-mm) sieve, the plus 3-in. (75-mm) material shall be removed prior to the determination of the particle-size characteristics and the liquid limit and plasticity index.

X4.4 The portion of the field sample finer than the 3-in. (75-mm) sieve shall be obtained as follows:

X4.4.1 Air dry and weigh the field sample.

X4.4.2 Separate the field sample into two fractions on a 3-in. (75-mm) sieve.

X4.4.3 Weigh the two fractions and compute the percentage of the plus 3-in. (75-mm) material in the field sample.

X4.5 Determine the particle-size distribution and liquid limit and plasticity index as follows (see 9.8 for when these tests are required):

X4.5.1 Thoroughly mix the fraction passing the 3-in. (75-mm) sieve.

X4.5.2 If the cumulative particle-size distribution including a hydrometer analysis is required, determine the particle-size distribution in accordance with Method D 422. See 9.7 for the set of sieves that is required.

X4.5.3 If the cumulative particle-size distribution without a hydrometer analysis is required, determine the particle-size distribution in accordance with Test Method D 1140 followed by Method C 136. See 9.7 for the set of sieves that is required.

X4.5.4 If the cumulative particle-size distribution is not required, determine the percent fines, percent sand, and percent gravel in the specimen in accordance with Test Method D 1140 followed by Method C 136 using a nest of sieves which shall include a No. 4 (4.75-mm) sieve and a No. 200 (75-μm) sieve.

X4.5.5 If required, determine the liquid limit and the plasticity index of the test specimen in accordance with Test Method D 4318.

## X5. RATIONALE

X5.1 Significant revisions were made to the standard which appeared as D 2487 – 83 from the previous version of D 2487 – 69 (1975). The changes are documented in the literature.

X5.2 Changes in this version from the last include the addition of 8.2 on classification of peat, the addition of 4.5 on classification of frozen soils, the addition of NOTE 6 for clarification of materials used to determine the plasticity index and liquid limit, and the addition of Appendix X5 on Rationale.

# F102
# *SOIL AND ROCK SAMPLE ACQUISITION*

# SOIL AND ROCK SAMPLE ACQUISITION

## 1.0 PURPOSE

The purpose of this procedure is to describe the handling of rock cores and subsurface soil samples collected during drilling operations. Surface soil sampling also is described.

## 2.0 SCOPE

The methods described in this SOP are applicable for the recovery of subsurface soil and rock samples acquired by coring operations or soil sampling techniques such as split-barrel sampling and thin-walled tube sampling. Procedures for the collection of surface soil samples also are discussed. This SOP does not discuss drilling techniques or well installation procedures. ASTM procedures for "Penetration Test and Split-Barrel Sampling of Soils," "Thin-Walled Tube Sampling of Soils," and "Diamond Core Drilling for Site Investigation" have been included as Attachments A through C, respectively.

## 3.0 DEFINITIONS

Thin-Walled Tube Sampler - A thin-walled metal tube (also called Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outer diameter (O.D.) and 18 to 54 inches length.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into unconsolidated materials using a drive weight mounted on the drilling string. A standard split-spoon sampler (used for performing Standard Penetration Tests) is two inches O.D. and 1-3/8-inches inner diameter (I.D.). This standard spoon is available in two common lengths providing either 20-inch or 26-inch internal longitudinal clearance for obtaining 18-inch or 24-inch long samples, respectively.

Grab Sample - An individual sample collected from a single location at a specific time or period of time generally not exceeding 15 minutes. Grab samples are associated with surface water, groundwater, wastewater, waste, contaminated surfaces, soil, and sediment sampling. Grab samples are typically used to characterize the media at a particular instant in time.

Composite Samples - A sample collected over time that typically consists of a series of discrete samples which are combined or "composited." Two types of composite samples are listed below:

- Areal Composite: A sample collected from individual grab samples collected on an areal or cross-sectional basis. Areal composites shall be made up of equal volumes of grab samples. Each grab sample shall be collected in an identical manner. Examples include sediment composites from quarter-point sampling of streams and soil samples from grid points.

- Vertical Composite: A sample collected from individual grab samples collected from a vertical cross section. Vertical composites shall be made up of equal volumes of grab samples. Each grab sample shall be collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes and estuaries.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that, where applicable, project-specific plans are in accordance with these procedures, or that other approved procedures are developed. Furthermore, the Project Manager is responsible for development of documentation of procedures which deviate from those presented herein.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the specific sampling techniques and equipment to be used, and documenting these in accordance with the Sampling and Analysis Plan. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures.

Drilling Inspector - It is the responsibility of the drilling inspector to follow these procedures, or to follow documented, project-specific procedures as directed by the Field Team Leader and/or the Project Manager. The Drilling Inspector is responsible for the proper acquisition of rock cores and subsurface soil samples.

Sampling Personnel - It is the responsibility of the field sampling personnel to follow these procedures, or to follow documented, project-specific procedures as directed by the Field Team Leader and/or the Project Manager. The sampling personnel are responsible for the proper acquisition of samples.

## 5.0 PROCEDURES

Subsurface soil and rock samples are used to characterize the three-dimensional subsurface stratigraphy. This characterization can indicate the potential for migration of contaminants from various sites. In addition, definition of the actual migration of contaminants can be obtained through chemical analysis of subsurface soil samples. Where the remedial activities may include in-situ treatment, or the excavation and removal of the contaminated soil, the depth and areal extent of contamination must be known as accurately as possible.

Surface soil samples serve to characterize the extent of surface contamination at various sites. These samples may be collected during initial site screening to determine gross contamination levels and levels of personal protection required as part of more intensive field sampling activities, to gather more detailed site data during design, or to determine the need for, or success of, cleanup actions.

Site construction activities may require that the engineering and physical properties of soil and rock be determined. Soil types, bearing strength, compressibility, permeability, plasticity, and moisture content are some of the geotechnical characteristics that may be determined by laboratory tests of soil samples. Rock quality, strength, stratigraphy, structure, etc. often are needed to design and construct deep foundations or remedial components.

### 5.1 Subsurface Soil Samples

This section discusses three methods for collecting subsurface soil samples: (1) split-spoon sampling; (2) shelby tube sampling; and, (3) bucket auger sampling. All three methods yield samples suitable for laboratory analysis. Copies of the ASTM procedures for split-spoon sampling and shelby-tube sampling are provided in Attachments A and B, respectively.

### 5.1.1 Split-Barrel (Split-Spoon) Sampling

The following procedures are to be used for split-spoon, geotechnical soil sampling:

1. Clean out the borehole to the desired sampling depth using equipment that will ensure that the material to be sampled is not disturbed by the operation.

2. Side-discharge or bottom-discharge bits are permissible. The process of jetting through the sampler and then sampling when the desired depth is reached shall not be permitted. Where casing is used, it may not be driven below the sampling elevation.

3. The two-inch O.D. split-barrel (not for geotech) sampler should be driven with blows from a 140-pound hammer falling 30 inches in accordance with ASTM D1586-84, Standard Penetration Test.

4. Repeat this operation at intervals not longer than 5 feet in homogeneous strata, or as specified in the Sampling and Analysis Plan.

5. Record on the Field Test Boring Record or field logbook the number of blows required to effect each six inches of penetration or fraction thereof. The first six inches is considered to be a seating drive. The sum of the number of blows required for the second and third six inches of penetration is termed the penetration resistance, N. If the sampler is driven less than 18 inches, the penetration resistance is that for the last one foot of penetration. (If less than one foot is penetrated, the logs shall state the number of blows and the fraction of one foot penetrated.) In cases where samples are driven 24 inches, the sum of second and third six-inch increments will be used to calculate the penetration resistance. (Refusal of the Standard Penetration Test will be noted as 50 blows over an interval equal to or less than 6 inches; the interval driven will be noted with the blow count.)

6. Bring the sampler to the surface and remove both ends and one half of the split-spoon such that the soil recovered rests in the remaining half of the barrel. Describe carefully the recovery (length), composition, structure, consistency, color, condition, etc. of the recovered soil according to SOP F101; then put into jars without ramming. Jars with samples not taken for chemical analysis should be tightly closed, to prevent evaporation of the soil moisture. Affix labels to the jar and complete Chain-of-Custody and other required sample data forms (see SOP F302). Protect samples against extreme temperature changes and breakage by placing them in appropriate cartons stored in a protected area.

In addition to collecting soils for geotechnical purposes, split-spoon sampling can be employed to obtain samples for environmental analytical analysis. The following procedures are to be used for split-spoon, environmental soil sampling:

1. Follow sample collection procedures 1 through 6 as outlined in Section 5.2.1.

2. After sample collection, remove the soil from the split-spoon sampler. Prior to filling laboratory containers, the soil sample should be mixed thoroughly as possible to ensure that the sample is as representative as possible of the sample interval. Soil samples for volatile organic compounds should not be mixed. Further, sample containers for volatile organic

compounds analyses should be filled completely without head space remaining in the container to minimize volatilization.

3. Record all pertinent sampling information such as soil description, sample depth, sample number, sample location, and time of sample collection in the Field Test Boring Record or field logbook. In addition, label, tag, and number the sample bottle(s).

4. Pack the samples for shipping (see SOP F301). Attach seal to the shipping package. Make sure that Chain-of-Custody Forms and Sample Request Forms are properly filled out and enclosed or attached (see SOP F302).

5. Decontaminate the split-spoon sample as described in SOP F501 and SOP F502. Replace disposable latex gloves between sample stations to prevent cross-contaminating samples.

For obtaining composite soil samples (see Section 3.0), a slightly modified approach is employed. Each individual discrete soil sample from the desired sample interval will be placed into a stainless-steel, decontaminated bowl (or other appropriate container) prior to filling the laboratory sample containers. Special care should be taken to cover the bowl between samples with aluminum foil to minimize volatilization. Immediately after obtaining soils from the desired sampling interval, the sample to be analyzed for volatile organic compounds (VOCs) should be collected. In the event that a composite sample is required, care should be taken to obtain a representative sampling of each sample interval. The remaining soils should be thoroughly mixed. Adequate mixing can be achieved by stirring in a circular fashion and occasionally turning the soils over. Once the remaining soils have been thoroughly combined, samples for analyses other than VOCs should be placed into the appropriate sampling containers.

### 5.1.2 Thin-Wall (Shelby Tube) Sampling

When it is desired to take undisturbed samples of soil for physical laboratory testing, thin-walled seamless tube samplers (Shelby tubes) will be used. The following method applies:

1. Clean out the hole to the sampling depth, being careful to minimize the chance for disturbance or contamination of the material to be sampled.

2. The use of bottom discharge bits or jetting through an open-tube sampler to clean out the hole shall not be allowed. Only side discharge bits are permitted.

3. Prior to inserting the tube sampler in the hole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out of the tube sampler during sample withdrawal and to maintain a suction within the tube to help retain the sample.

4. With the sampling tube resting on the bottom of the hole and the water level in the boring at the natural groundwater level or above, push the tube into the soil by a continuous and rapid motion, without impacting or twisting. In no case shall the tube be pushed further than the length provided for the soil sample. Allow a free space in the tube for cuttings and sludge.

5. After pushing the tube, the sample should sit 5 to 15 minutes prior to removal. Immediately before removal, the sample must be sheared by rotating the rods with a pipe wrench a minimum of two revolutions.

6. Upon removal of the sampler tube from the hole, measure the length of sample in the tube and also the length penetrated. Remove disturbed material in the upper end of the tube and measure the length of sample again. After removing at least an inch of soil, from the lower end and after inserting an impervious disk, seal both ends of the tube with at least a 1/2-inch thickness of wax applied in a way that will prevent the wax from entering the sample. Newspaper or other types of filler must be placed in voids at either end of the sampler prior to sealing with wax. Place plastic caps on the ends of the sampler, tape them into place and then dip the ends in wax to seal them.

7. Affix labels to the tubes and record sample number, depth, penetration, and recovery length on the label. Mark the same information and "up" direction on the tube with indelible ink, and indicate the top of the sample. Complete chain-of-custody and other required forms (see SOP F302). Do not allow tubes to freeze, and store the samples vertically (with the same orientation they had in the ground, i.e., top of sample is up) in a cool place out of the sun at all times. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

8. From soil removed from the ends of the tube, make a careful description using the methods presented in SOP F101.

9. When thin-wall tube samplers are used to collect soil for certain chemical analyses, it may be necessary to avoid using wax, newspaper, or other fillers.

Thin-walled undisturbed tube samplers are restricted in their usage by the consistency of the soil to be sampled. Often very loose and/or wet samples cannot be retrieved by the samplers, and soils with a consistency in excess of very stiff cannot be penetrated by the sampler. Other appropriate devices can be used in conjunction with the tube samplers to obtain undisturbed samples of stiff soils. Using these devices normally increases sampling costs and, therefore, their use should be weighed against the increased cost and the need for an undisturbed sample. In any case, if a sample cannot be obtained with a tube sampler, an attempt should be made with a split-spoon sampler at the same depth so that at least one sample can be obtained for classification purposes.

### 5.1.3 Bucket (Hand) Auger Sampling

Hand augering is the most common manual method used to collect subsurface samples. Typically, 4-inch auger buckets with cutting heads are pushed and twisted into the ground and removed as the buckets are filled. The auger holes are advanced one bucket at a time. The practical depth of investigation using a hand auger is related to the material being sampled. In sands, augering is usually easily accomplished, but the depth of investigation is controlled by the depth at which sands begin to cave. At this point, auger holes usually begin to collapse and cannot practically be advanced to lower depths, and further samples, if required, must be collected using some type of pushed or driven device. Hand augering may also become difficult in tight clays or cemented sands. At depths approaching 20 feet, torquing of hand auger extensions becomes so severe that in resistant materials powered methods must be used.

When a vertical sampling interval has been established, one auger bucket is used to advance the auger hole to the first desired sampling depth. If the sample at this location is to be a vertical composite of all intervals, the same bucket may be used to advance the hole, as well collect subsequent samples in the same hole. However, if discrete grab samples are to be collected to characterize each depth, a decontaminated bucket must be placed on the end of the auger extension immediately prior to collecting the next sample. The top several inches of soil should be removed from the bucket to minimize the chances of cross-contamination of the sample from fall-in of material from the upper portions of the hole. The bucket auger should be decontaminated between samples as outlined in SOP F502.

In addition to hand augering, powered augers can be used to advance a boring for subsurface soil collection. However, this type of equipment is technically a sampling aid and not a sampling device, and 20 to 25 feet is the typical lower depth range for this equipment. It is used to advance a hole to the required sample depth, at which point a hand auger is usually used to collect the sample.

## 5.2 Surface Soil Samples

Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. For loosely packed surface soils, stainless steel (organic analyses) or plastic (inorganic analyses) scoops or trowels, can be used to collect representative samples. For densely packed soils or deeper soil samples, a hand or power soil auger may be used.

The following methods are to be used:

1. Use a soil auger for deep samples (greater than 12 inches) or a scoop or trowel for surface samples. Remove debris, rocks, twigs, and vegetation before collecting the sample.

2. Immediately transfer the sample to the appropriate sample container. Attach a label and identification tag. Record all required information in the field logbook (SOP F303) and on the sample log sheet, chain-of-custody record (SOP F302), and other required forms.

3. Classify and record a description of the sample, as discussed in SOP F101. Descriptions for surface soil samples should be recorded in the field logbook; descriptions for soil samples collected with power or hand augers shall be recorded on a Field Test Boring Record.

4. Store the sampling utensil in a plastic bag until decontamination or disposal. Use a new or freshly-decontaminated sampling utensil for each sample taken.

5. Pack and ship as described in SOP F301.

6. Mark the location with a numbered stake if possible and locate sample points on a sketch of the site or on a sketch in the field logbook.

7. When a representative composited sample is to be prepared (e.g., samples taken from a gridded area or from several different depths), it is best to composite individual samples in the laboratory where they can be more precisely composited on a weight or volume basis. If this is not possible, the individual samples (all of equal volume, i.e., the sample bottles should be full) should be placed in a stainless steel bucket (or other appropriate container), mixed thoroughly using a decontaminated stainless steel spatula or trowel, and a composite

sample collected. In some cases, as delineated in project-specific sampling and analysis plans, laboratory compositing of the samples may be more appropriate than field compositing. Samples to be analyzed for parameters sensitive to volatilization should be composited and placed into the appropriate sample bottles immediately upon collection.

## 5.3 Rock Cores

Once rock coring has been completed and the core recovered, the rock core must be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery, as well as the rock quality designation (RQD) (see SOP F101). If split-barrels are used, the core may be measured and classified in the split barrel after opening and then transferred to a core box.

Each core shall be described and classified on a Field Test Boring Record using a uniform system as presented in SOP F101. If moisture content will be determined or if it is desirable to prevent drying (e.g., to prevent shrinkage of hydrated formations) or oxidation of the core, the core must be wrapped in plastic sleeves immediately after logging. Each plastic sleeve shall be labeled with indelible ink. The boring number, run number and the footage represented in each sleeve shall be included, as well as the top and bottom of the core run.

After sampling, rock cores must be placed in the sequence of recovery in wooden or plastic core boxes provided by the drilling contractor. Rock cores from different borings shall not be placed in the same core box. The core boxes should be constructed to accommodate 10 to 20 linear feet of core and should be constructed with hinged tops secured with screws, and a latch (usually a hook and eye) to keep the top securely fastened. Wood partitions shall be placed at the end of each core run and between rows. The depth from the surface of the boring to the top and bottom of the drill run and the run number shall be marked on the wooden partitions with indelible ink. The order of placing cores shall be the same in all core boxes. The top of each core obtained should be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, any empty space in a row shall be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core-box lid shall be marked by indelible ink to show all pertinent data pertaining to the box's contents. At a minimum, the following information must be included:

- Project name
- Date
- Boring number
- Footage (depths)
- Run number(s)
- Recovery
- Rock Quality Designation (RQD)
- Box number (x of x)

It is also useful to draw a large diagram of the core in the box, on the inside of the box top. This provides more room for elevations, run numbers, recoveries, comments, etc., than could be entered on the upper edges of partitions or spaces in the core box.

For easy retrieval when core boxes are stacked, the sides and ends of the box should also be labeled and include project name, boring number, top and bottom depths of core and box number.

Due to the weight of the core, a filled core box should always be handled by two people. Core boxes stored on site should be protected from the weather. The core boxes should be removed from the site in a careful manner as soon as possible. Exposure to extreme heat or cold should be avoided whenever possible. Arrangements should be made to dispose of or return the core samples to the client for completion of the project.

## 6.0 QUALITY ASSURANCE RECORDS

Where applicable, Field Test Boring Records and Test Boring Records will serve as the quality assurance records for subsurface soil samples, rock cores and near surface soil samples collected with a hand or power auger. Observations shall be recorded in the Field Logbook as described in SOP F303. Chain-of-Custody records shall be completed for samples collected for laboratory analysis as described in SOP F101 and SOP F302.

## 7.0 REFERENCES

1. American Society for Testing and Materials, 1987. Standard Method for Penetration Test and Split-Barrel Sampling of Soils. ASTM Method D1586-84, Annual Book of Standards, ASTM, Philadelphia, Pennsylvania.

2. American Society for Testing and Materials, 1987. Standard Practice for Thin-Walled Tube Sampling of Soils. Method D1587-83, Annual Book of Standards, ASTM, Philadelphia, Pennsylvania.

3. American Society for Testing and Materials, 1987. Standard Practice for Diamond Core Drilling for Site Investigation. Method D2113-83 (1987), Annual Book of Standards ASTM, Philadelphia, Pennsylvania.

4. U. S. EPA, 1991. Standard Operating Procedures and Quality Assurance Manual. Environmental Compliance Branch, U. S. EPA, Environmental Services Division, Athens, Georgia.

# ATTACHMENT A

## ASTM D1586-84
## STANDARD METHOD FOR PENETRATION TEST AND SPLIT-BARREL SAMPLING OF SOILS

Designation: D 1586 – 84



# Standard Method for Penetration Test and Split-Barrel Sampling of Soils[1]

This standard is issued under the fixed designation D 1586; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This method has been approved for use by agencies of the Department of Defense and for listing in the DOD Index of Specifications and Standards.*

## 1. Scope

1.1 This method describes the procedure, generally known as the Standard Penetration Test (SPT), for driving a split-barrel sampler to obtain a representative soil sample and a measure of the resistance of the soil to penetration of the sampler.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific precautionary statement, see 5.4.1.

1.3 The values stated in inch-pound units are to be regarded as the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 2487 Test Method for Classification of Soils for Engineering Purposes[2]

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[2]

D 4220 Practices for Preserving and Transporting Soil Samples[2]

## 3. Descriptions of Terms Specific to This Standard

3.1 *anvil*—that portion of the drive-weight assembly which the hammer strikes and through which the hammer energy passes into the drill rods.

3.2 *cathead*—the rotating drum or windlass in the rope-cathead lift system around which the operator wraps a rope to lift and drop the hammer by successively tightening and loosening the rope turns around the drum.

3.3 *drill rods*—rods used to transmit downward force and torque to the drill bit while drilling a borehole.

3.4 *drive-weight assembly*—a device consisting of the hammer, hammer fall guide, the anvil, and any hammer drop system.

3.5 *hammer*—that portion of the drive-weight assembly consisting of the 140 ± 2 lb (63.5 ± 1 kg) impact weight which is successively lifted and dropped to provide the energy that accomplishes the sampling and penetration.

3.6 *hammer drop system*—that portion of the drive-weight assembly by which the operator accomplishes the lifting and dropping of the hammer to produce the blow.

3.7 *hammer fall guide*—that part of the drive-weight assembly used to guide the fall of the hammer.

3.8 *N-value*—the blowcount representation of the penetration resistance of the soil. The *N*-value, reported in blows per foot, equals the sum of the number of blows required to drive the sampler over the depth interval of 6 to 18 in. (150 to 450 mm) (see 7.3).

3.9 $\Delta N$—the number of blows obtained from each of the 6-in. (150-mm) intervals of sampler penetration (see 7.3).

3.10 *number of rope turns*—the total contact angle between the rope and the cathead at the beginning of the operator's rope slackening to drop the hammer, divided by 360° (see Fig. 1).

3.11 *sampling rods*—rods that connect the drive-weight assembly to the sampler. Drill rods are often used for this purpose.

3.12 *SPT*—abbreviation for Standard Penetration Test, a term by which engineers commonly refer to this method.

## 4. Significance and Use

4.1 This method provides a soil sample for identification purposes and for laboratory tests appropriate for soil obtained from a sampler that may produce large shear strain disturbance in the sample.

4.2 This method is used extensively in a great variety of geotechnical exploration projects. Many local correlations and widely published correlations which relate SPT blowcount, or *N*-value, and the engineering behavior of earthworks and foundations are available.

## 5. Apparatus

5.1 *Drilling Equipment*—Any drilling equipment that provides at the time of sampling a suitably clean open hole before insertion of the sampler and ensures that the penetration test is performed on undisturbed soil shall be acceptable. The following pieces of equipment have proven to be suitable for advancing a borehole in some subsurface conditions.

5.1.1 *Drag, Chopping, and Fishtail Bits*, less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm) in diameter may be used in conjuction with open-hole rotary drilling or casing-advancement drilling methods. To avoid disturbance of the underlying soil, bottom discharge bits are not permitted; only side discharge bits are permitted.

5.1.2 *Roller-Cone Bits*, less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm) in diameter may be used in

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[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Investigations.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

**ATTACHMENT B**

**ASTM D1587-83**
**STANDARD PRACTICE FOR THIN-WALLED TUBE SAMPLING OF SOILS**

Designation: D 1587 – 83



# Standard Practice for Thin-Walled Tube Sampling of Soils[1]

This standard is issued under the fixed designation D 1587; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This practice has been approved for use by agencies of the Department of Defense and for listing in the DOD Index of Specifications and Standards.*

## 1. Scope

1.1 This practice covers a procedure for using a thin-walled metal tube to recover relatively undisturbed soil samples suitable for laboratory tests of structural properties. Thin-walled tubes used in piston, plug, or rotary-type samplers, such as the Denison or Pitcher, must comply with the portions of this practice which describe the thin-walled tubes (5.3).

NOTE 1—This practice does not apply to liners used within the above samplers.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[2]

D 3550 Practice for Ring-Lined Barrel Sampling of Soils[2]

D 4220 Practices for Preserving and Transporting Soil Samples[2]

## Summary of Practice

3.1 A relatively undisturbed sample is obtained by pressing a thin-walled metal tube into the in-situ soil, removing the soil-filled tube, and sealing the ends to prevent the soil from being disturbed or losing moisture.

## 4. Significance and Use

4.1 This practice, or Practice D 3550, is used when it is necessary to obtain a relatively undisturbed specimen suitable for laboratory tests of structural properties or other tests that might be influenced by soil disturbance.

## 5. Apparatus

5.1 *Drilling Equipment*—Any drilling equipment may be used that provides a reasonably clean hole; that does not disturb the soil to be sampled; and that does not hinder the penetration of the thin-walled sampler. Open borehole diameter and the inside diameter of driven casing or hollow stem auger shall not exceed 3.5 times the outside diameter of the thin-walled tube.

5.2 *Sampler Insertion Equipment*, shall be adequate to provide a relatively rapid continuous penetration force. For hard formations it may be necessary, although not recommended, to drive the thin-walled tube sampler.

5.3 *Thin-Walled Tubes*, should be manufactured as shown in Fig. 1. They should have an outside diameter of 2 to 5 in. and be made of metal having adequate strength for use in the soil and formation intended. Tubes shall be clean and free of all surface irregularities including projecting weld seams.

5.3.1 *Length of Tubes*—See Table 1 and 6.4.

5.3.2 *Tolerances*, shall be within the limits shown in Table 2.

5.3.3 *Inside Clearance Ratio*, should be 1 % or as specified by the engineer or geologist for the soil and formation to be sampled. Generally, the inside clearance ratio used should increase with the increase in plasticity of the soil being sampled. See Fig. 1 for definition of inside clearance ratio.

5.3.4 *Corrosion Protection*—Corrosion, whether from galvanic or chemical reaction, can damage or destroy both the thin-walled tube and the sample. Severity of damage is a function of time as well as interaction between the sample and the tube. Thin-walled tubes should have some form of protective coating. Tubes which will contain samples for more than 72 h shall be coated. The type of coating to be used may vary depending upon the material to be sampled. Coatings may include a light coat of lubricating oil, lacquer, epoxy, Teflon, and others. Type of coating must be specified by the engineer or geologist if storage will exceed 72 h. Plating of the tubes or alternate base metals may be specified by the engineer or geologist.

5.4 *Sampler Head*, serves to couple the thin-walled tube to the insertion equipment and, together with the thin-walled tube, comprises the thin-walled tube sampler. The sampler head shall contain a suitable check valve and a venting area to the outside equal to or greater than the area through the check valve. Attachment of the head to the tube shall be concentric and coaxial to assure uniform application of force to the tube by the sampler insertion equipment.

## 6. Procedure

6.1 Clean out the borehole to sampling elevation using whatever method is preferred that will ensure the material to be sampled is not disturbed. If groundwater is encountered, maintain the liquid level in the borehole at or above ground water level during the sampling operation.

6.2 Bottom discharge bits are not permitted. Side discharge bits may be used, with caution. Jetting through an open-tube sampler to clean out the borehole to sampling elevation is not permitted. Remove loose material from the center of a casing or hollow stem auger as carefully as

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Investigations.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

ATTACHMENT C

ASTM D2113-83 (1987)
STANDARD PRACTICE FOR DIAMOND CORE DRILLING FOR SITE INVESTIGATION

Designation: D 2113 – 83 (Reapproved 1987)

# Standard Practice for Diamond Core Drilling for Site Investigation[1]

This standard is issued under the fixed designation D 2113; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice describes equipment and procedures for diamond core drilling to secure core samples of rock and some soils that are too hard to sample by soil-sampling methods. This method is described in the context of obtaining data for foundation design and geotechnical engineering purposes rather than for mineral and mining exploration.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1586 Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 3550 Practice for Ring-Lined Barrel Sampling of Soils[2]

## 3. Significance and Use

3.1 This practice is used to obtain core specimens of [illegible]or quality that reflect the in-situ conditions of the [illegible]al and structure and which are suitable for standard physical-properties tests and structural-integrity determination.

## 4. Apparatus

4.1 *Drilling Machine*, capable of providing rotation, feed, and retraction by hydraulic or mechanical means to the drill rods.

4.2 *Fluid Pump or Air Compressor*, capable of delivering sufficient volume and pressure for the diameter and depth of hole to be drilled.

4.3 *Core barrels*, as required:

4.3.1 *Single Tube Type, WG Design*, consisting of a hollow steel tube, with a head at one end threaded for drill rod, and a threaded connection for a reaming shell and core bit at the other end. A core lifter, or retainer located within the core bit is normal, but may be omitted at the discretion of the geologist or engineer.

4.3.2 *Double Tube, Swivel-Type, WG Design*—An assembly of two concentric steel tubes joined and supported at the upper end by means of a ball or roller-bearing swivel arranged to permit rotation of the outer tube without causing rotation of the inner tube. The upper end of the outer tube, or removable head, is threaded for drill rod. A threaded connection is provided on the lower end of the outer tube for a reaming shell and core bit. A core lifter located within the core bit is normal but may be omitted at the discretion of the geologist or engineer.

4.3.3 *Double-Tube, Swivel-Type, WT Design*, is essentially the same as the double tube, swivel-type, WG design, except that the WT design has thinner tube walls, a reduced annular area between the tubes, and takes a larger core from the same diameter bore hole. The core lifter is located within the core bit.

4.3.4 *Double Tube, Swivel Type, WM Design*, is similar to the double tube, swivel-type, WG design, except that the inner tube is threaded at its lower end to receive a core lifter case that effectively extends the inner tube well into the core bit, thus minimizing exposure of the core to the drilling fluid. A core lifter is contained within the core lifter case on the inner tube.

4.3.5 *Double Tube Swivel-Type, Large-Diameter Design*, is similar to the double tube, swivel-type, WM design, with the addition of a ball valve, to control fluid flow, in all three available sizes and the addition of a sludge barrel, to catch heavy cuttings, on the two larger sizes. The large-diameter design double tube, swivel-type, core barrels are available in three core per hole sizes as follows: 2¼ in. (69.85 mm) by 3¼ in. (98.43 mm), 4 in. (101.6 mm) by 5½ in. (139.7 mm), and 6 in. (152.4 mm) by 7¾ in. (196.85 mm). Their use is generally reserved for very detailed investigative work or where other methods do not yield adequate recovery.

4.3.6 *Double Tube, Swivel-Type, Retrievable Inner-Tube Method*, in which the core-laden inner-tube assembly is retrieved to the surface and an empty inner-tube assembly returned to the face of the borehole through the matching large-bore drill rods without need for withdrawal and replacement of the drill rods in the borehole. The inner-tube assembly consists of an inner tube with removable core lifter case and core lifter at one end and a removable inner-tube head, swivel bearing, suspension adjustment, and latching device with release mechanism on the opposite end. The inner-tube latching device locks into a complementary recess in the wall of the outer tube such that the outer tube may be rotated without causing rotation of the inner tube and such that the latch may be actuated and the inner-tube assembly transported by appropriate surface control. The outer tube is threaded for the matching, large-bore drill rod and internally configured to receive the inner-tube latching device at one end and threaded for a reaming shell and bit, or bit only, at the other end.

4.4 *Longitudinally Split Inner Tubes*—As opposed to conventional cylindrical inner tubes, allow inspection of, and access to, the core by simply removing one of the two halves. They are not standardized but are available for most core barrels including many of the retrievable inner-tube types.

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[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Investigations.

Current edition approved June 24, 1983. Published August 1983. Originally published as D 2113 – 62 T. Last previous edition D 2113 – 70 (1976).

[2] *Annual Book of ASTM Standards*, Vol 04.08.

4.5 *Core Bits*—Core bits shall be surface set with diamonds, impregnated with small diamond particles, inserted with tungsten carbide slugs, or strips, hard-faced with various hard surfacing materials or furnished in saw-tooth form, all as appropriate to the formation being cored and with concurrence of the geologist or engineer. Bit matrix material, crown shape, water-way type, location and number of water ways, diamond size and carat weight, and bit facing materials shall be for general purpose use unless otherwise approved by the geologist or engineer. Nominal size of some bits is shown in Table 1.

NOTE 1—Size designation (letter symbols) used throughout the text and in Tables 1, 2, and 3 are those standardized by the Diamond Core Drill Manufacturers' Assoc. (DCDMA). Inch dimensions in the tables have been rounded to the nearest hundredth of an inch.

4.6 *Reaming Shells*, shall be surface set with diamonds, impregnated with small diamond particles, inserted with tungsten carbide strips or slugs, hard faced with various types of hard surfacing materials, or furnished blank, all as appropriate to the formation being cored.

4.7 *Core Lifters*—Core lifters of the split-ring type, either thin or hard-faced, shall be furnished and maintained, along with core-lifter cases or inner-tube extensions or inner-tube shoes, in good condition. Basket or finger-type lifters, together with any necessary adapters, shall be on the job and available for use with each core barrel if so directed by the geologist or engineer.

4.8 *Casings:*

4.8.1 *Drive Pipe or Drive Casing*, shall be standard weight (schedule 40), extra-heavy (schedule 80), double extra-heavy (schedule 160) pipe or W-design flush-joint casing as required by the nature of the overburden or the placement method. Drive pipe or W-design casing shall be of sufficient diameter to pass the largest core barrel to be used, and it shall be driven to bed rock or to firm seating at an elevation below water-sensitive formation. A hardened drive shoe is to be used as a cutting edge and thread protection device on the bottom of the drive pipe or casing. The drive shoe inside diameter shall be large enough to pass the tools intended for use, and the shoe and pipe or casing shall be free from burrs or obstructions.

4.8.2 *Casing*—When necessary to case through formations already penetrated by the borehole or when no drive casing has been set, auxiliary casing shall be provided to fit inside the borehole to allow use of the next smaller core barrel. Standard sizes of telescoping casing are shown in Table 2. Casing bits have an obstruction in their interior and will not pass the next smaller casing size. Use a casing shoe if additional telescoping is anticipated.

4.8.3 *Casing Liner*—Plastic pipe or sheet-metal pipe may be used to line an existing large-diameter casing. Liners, so used, should not be driven, and care should be taken to maintain true alignment throughout the length of the liner.

4.8.4 *Hollow Stem Auger*—Hollow stem auger may be used as casing for coring.

4.9 *Drill Rods:*

4.9.1 *Drill Rods of Tubular Steel Construction* are normally used to transmit feed, rotation, and retraction forces from the drilling machine to the core barrel. Drill-rod sizes that are presently standardized are shown in Table 3.

4.9.2 Large bore drill rods used with retrievable inner-tube core barrels are not standardized. Drill rods used with retrievable inner-tube core barrels should be those manufactured by the core-barrel manufacturer specifically for the core barrel.

4.9.3 *Composite Drill Rods* are specifically constructed from two or more materials intended to provide specific properties such as light weight or electrical nonconductivity.

4.9.4 *Nonmagnetic Drill Rods* are manufactured of nonferrous materials such as aluminum or brass and are used primarily for hole survey work. Some nonmagnetic rods have left-hand threads in order to further their value in survey work. No standard exists for nonmagnetic rods.

4.10 *Auxiliary Equipment*, shall be furnished as required by the work and shall include: roller rock bits, drag bits, chopping bits, boulder busters, fishtail bits, pipe wrenches, core barrel wrenches, lubrication equipment, core boxes, and marking devices. Other recommended equipment includes:

TABLE 1 Core Bit Sizes

| Size Designation | Outside Diameter | | Inside Diameter | |
|---|---|---|---|---|
| | in. | mm | in. | mm |
| RWT | 1.16 | 29.5 | 0.375 | 18.7 |
| EWT | 1.47 | 37.3 | 0.905 | 22.9 |
| EWG, EWM | 1.47 | 37.3 | 0.845 | 21.4 |
| AWT | 1.88 | 47.8 | 1.281 | 32.5 |
| AWG, AWM | 1.88 | 47.8 | 1.185 | 30.0 |
| BWT | 2.35 | 59.5 | 1.750 | 44.6 |
| BWG, BWM | 2.35 | 59.5 | 1.655 | 42.0 |
| NWT | 2.97 | 75.3 | 2.313 | 58.7 |
| NWG, NWM | 2.97 | 75.3 | 2.155 | 54.7 |
| 2¾ × 3⅞ | 3.84 | 97.5 | 2.69 | 68.3 |
| HWT | 3.89 | 98.8 | 3.187 | 80.9 |
| HWG. . . . | 3.89 | 98.8 | 3.000 | 76.2 |
| 4 × 5½ | 5.44 | 138.0 | 3.97 | 100.8 |
| 6 × 7¾ | 7.66 | 194.4 | 5.97 | 151.8 |

TABLE 2 Casing Sizes

| Size Designation | Outside Diameter | | Inside Diameter | | Threads per in. | Will Fit Hole Drilled with Core Bit Size |
|---|---|---|---|---|---|---|
| | in. | mm | in. | mm | | |
| RW | 1.144 | 36.5 | 1.19 | 30.1 | 5 | EWT, EWG, EWM |
| EW | 1.81 | 46.0 | 1.50 | 38.1 | 4 | AWT, AWG, AWM |
| AW | 2.25 | 57.1 | 1.91 | 48.4 | 4 | BWT, BWG, BWM |
| BW | 2.86 | 73.0 | 2.38 | 60.3 | 4 | NWT, NWG, NWM |
| NW | 3.50 | 88.9 | 3.00 | 76.2 | 4 | HWT, HWG |
| HW | 4.50 | 114.3 | 4.00 | 101.6 | 4 | 4 × 5½ |
| PW | 5.50 | 139.7 | 5.00 | 127.0 | 3 | 6 × 7¾ |
| SW | 6.63 | 168.2 | 6.00 | 152.4 | 3 | 6 × 7¾ |
| UW | 7.63 | 193.6 | 7.00 | 177.8 | 2 | . . . |
| ZW | 8.63 | 219.0 | 8.00 | 203.2 | 2 | . . . |

TABLE 3 Drill Rods

| Designation | Rod and Coupling Outside Diameter | | Rod Inside Diameter | | Coupling Bore, Threads | | |
|---|---|---|---|---|---|---|---|
| | in. | mm | in. | mm | in. | mm | per in. |
| RW | 1.09 | 27.7 | 0.72 | 18.2 | 0.41 | 10.3 | 4 |
| EW | 1.38 | 34.9 | 1.00 | 25.4 | 0.44 | 11.1 | 3 |
| AW | 1.72 | 43.6 | 1.34 | 34.1 | 0.63 | 16.8 | 3 |
| BW | 2.13 | 53.9 | 1.75 | 44.4 | 0.75 | 19.0 | 3 |
| NW | 2.63 | 66.6 | 2.25 | 57.1 | 1.38 | 34.9 | 3 |
| HW | 3.50 | 88.9 | 3.06 | 77.7 | 2.38 | 60.3 | 3 |

core splitter, rod wicking, pump-out tools or extruders, and hand sieve or strainer.

## 5. Transportation and Storage of Core Containers

5.1 *Core Boxes,* shall be constructed of wood or other durable material for the protection and storage of cores while enroute from the drill site to the laboratory or other processing point. All core boxes shall be provided with longitudinal separators and recovered cores shall be laid out as a book would read, from left to right and top to bottom, within the longitudinal separators. Spacer blocks or plugs shall be marked and inserted into the core column within the separators to indicate the beginning of each coring run. The beginning point of storage in each core box is the upper left-hand corner. The upper left-hand corner of a hinged core box is the left corner when the hinge is on the far side of the box and the box is right-side up. All hinged core boxes must be permanently marked on the outside to indicate the top and the bottom. All other core boxes must be permanently marked on the outside to indicate the top and the bottom and additionally, must be permanently marked internally to the upper-left corner of the bottom with the letters a splotch of red paint not less than 1 in.[2] Lid or cover fitting(s) for core boxes must be of such quality as to ensure against mix up of the core in the event of impact or upsetting of the core box during transportation.

5.2 Transportation of cores from the drill site to the laboratory or other processing point shall be in durable core boxes so padded or suspended as to be isolated from shock or impact transmitted to the transporter by rough terrain or careless operation.

5.3 Storage of cores, after initial testing or inspection at the laboratory or other processing point, may be in cardboard or similar less costly boxes provided all layout and marking requirements as specified in 5.1 are followed. Additional spacer blocks or plugs shall be added if necessary at time of storage to explain missing core. Cores shall be stored for a period of time specified by the engineer but should not normally be discarded prior to completion of the project for which they were taken.

## Procedure

6.1 Use core-drilling procedures when formations are encountered that are too hard to be sampled by soil-sampling methods. A 1-in. (25.4-mm) or less penetration for 50 blows in accordance with Method D 1586 or other criteria established by the geologist or engineer, shall indicate that sampling methods are not applicable.

6.1.1 Seat the casing on bedrock or in a firm formation to prevent raveling of the borehole and to prevent loss of drilling fluid. Level the surface of the rock or hard formation at the bottom of the casing when necessary, using the appropriate bits. Casing may be omitted if the borehole will stand open without the casing.

6.1.2 Begin the core drilling using an N-size double-tube swivel-type core barrel or other size or type approved by the engineer. Continue core drilling until core blockage occurs or until the net length of the core barrel has been drilled in. Remove the core barrel from the hole and disassemble it as necessary to remove the core. Reassemble the core barrel and return it to the hole. Resume coring.

6.1.3 Place the recovered core in the core box with the upper (surface) end of the core at the upper-left corner of the core box as described in 5.1. Continue boxing core with appropriate markings, spacers, and blocks as described in 5.1. Wrap soft or friable cores or those which change materially upon drying in plastic film or seal in wax, or both, when such treatment is required by the engineer. Use spacer blocks or slugs properly marked to indicate any noticeable gap in recovered core which might indicate a change or void in the formation. Fit fracture, bedded, or jointed pieces of core together as they naturally occurred.

6.1.4 Stop the core drilling when soft materials are encountered that produce less than 50 % recovery. If necessary, secure samples of soft materials in accordance with the procedures described in Method D 1586, Practice D 1587, or Practice D 3550, or by any other method acceptable to the geologist or engineer. Resume diamond core drilling when refusal materials as described in 6.1 are again encountered.

6.2 Subsurface structure, including the dip of strata, the occurrence of seams, fissures, cavities, and broken areas are among the most important items to be detected and described. Take special care to obtain and record information about these features. If conditions prevent the continued advance of the core drilling, the hole should be cemented and redrilled, or reamed and cased, or cased and advanced with the next smaller-size core barrel, as required by the geologist or engineer.

6.3 Drilling mud or grouting techniques must be approved by the geologist or engineer prior to their use in the borehole.

6.4 *Compatibility of Equipment:*

6.4.1 Whenever possible, core barrels and drill rods should be selected from the same letter-size designation to ensure maximum efficiency. See Tables 1 and 3.

6.4.2 Never use a combination of pump, drill rod, and core barrel that yields a clear-water up-hole velocity of less than 120 ft/min.

6.4.3 Never use a combination of air compressor, drill rod, and core barrel that yields a clear-air up-hole velocity of less than 3000 ft/min.

7. Boring Log

7.1 The boring log shall include the following:

7.1.1 Project identification, boring number, location, date boring began, date boring completed, and driller's name.

7.1.2 Elevation of the ground surface.

7.1.3 Elevation of or depth to ground water and raising or lowering of level including the dates and the times measured.

7.1.4 Elevations or depths at which drilling fluid return was lost.

7.1.5 Size, type, and design of core barrel used. Size, type, and set of core bit and reaming shell used. Size, type, and length of all casing used. Description of any movements of the casing.

7.1.6 Length of each core run and the length or percentage, or both, of the core recovered.

7.1.7 Geologist's or engineer's description of the formation recovered in each run.

7.1.8 Driller's description, if no engineer or geologist is present, of the formation recovered in each run.

7.1.9 Subsurface structure description, including dip of strata and jointing, cavities, fissures, and any other observations made by the geologist or engineer that could yield information regarding the formation.

7.1.10 Depth, thickness, and apparent nature of the filling of each cavity or soft seam encountered, including opinions gained from the feel or appearance of the inside of the inner tube when core is lost. Record opinions as such.

7.1.11 Any change in the character of the drilling fluid or drilling fluid return.

7.1.12 Tidal and current information when the borehole is sufficiently close to a body of water to be affected.

7.1.13 Drilling time in minutes per foot and bit pressure in pound-force per square inch gage when applicable.

7.1.14 Notations of character of drilling, that is, soft, slow, easy, smooth, etc.

8. Precision and Bias

8.1 This practice does not produce numerical data; therefore, a precision and bias statement is not applicable.

NOTE 2—Inclusion of the following tables and use of letter symbols in the foregoing text is not intended to limit the practice to use of DCDMA tools. The table and text references are included as a convenience to the user since the vast majority of tools in use do meet DCDMA dimensional standards. Similar equipment of approximately equal size on the metric standard system is acceptable unless otherwise stipulated by the engineer or geologist.

# F103
# *MONITOR WELL INSTALLATION*

# MONITORING WELL INSTALLATION

## 1.0 PURPOSE

The purpose of this procedure is to provide general guidance and reference material regarding the installation of monitoring wells at various sites.

## 2.0 SCOPE

This SOP describes the methods of installing a groundwater monitoring well, and creating a Monitoring Well Installation Record. This SOP does not discuss drilling, soil sampling, borehole logging or related activities. These other activities are discussed in SOPs F102 and F101 entitled Soil and Rock Sample Acquisition, and Borehole and Sample Logging, respectively.

## 3.0 DEFINITIONS

Monitoring Well - A monitoring well is a well which is properly screened, cased, and sealed to intercept a discrete zone of the subsurface, and is capable of providing a groundwater level and sample representative of the zone being monitored.

Piezometer - A piezometer is a pipe or tube inserted into an aquifer or other water-bearing zone, open to water flow at the bottom, open to the atmosphere at the top, and used to measure water level elevations. Piezometers are not used for the collection of groundwater quality samples or aquifer characteristic data other than water level elevations.

Drive Point - A monitoring well which includes a screen casing and hardened point fabricated from stainless steel that is driven into the soil to complete the well. The drive point can also be installed by hand augering to try to formulate a sand pack around the screen.

## 4.0 RESPONSIBILITIES

Project Manager - It is the responsibility of the Project Manager to ensure that field personnel installing monitoring wells are familiar with these procedures. The Project Manager also is responsible for ensuring that all appropriate documents (e.g., test boring logs, monitoring well construction logs, etc.) have been correctly and completely filled out by the drilling inspector.

Field Team Leader - The Field Team Leader is responsible for the overall supervision of all drilling, boring and well installation activities, and for ensuring that the well is completely and correctly installed and logged. The Field Team Leader also is responsible for ensuring that all drilling inspectors have been briefed on these procedures. The Field Team Leader is responsible to provide copies of the well construction logs and field log books to the Project File via the Project Manager on a weekly basis, unless otherwise specified by the Project Manager.

Drilling Inspector (Site Geologist) - The Drilling Inspector or Site Geologist is responsible for the direct supervision of drilling and well installation activities. It is the Drilling Inspector's responsibility to record details of the well installation, document subsurface conditions, complete the appropriate forms, supervise the drilling crew (or drilling supervisor), and record quantities of the drillers billable labor and materials.

## 5.0 PROCEDURES

The objectives for the use of each monitoring well and of the entire array of wells must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, may require different types of construction. During all phases of the well design (both office and field), attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used.

The objectives for installing monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for groundwater contamination.
- Determining aquifer characteristics (e.g., hydraulic conductivity).
- Facilitating site remediation via injection or recovery.

In cases where only the groundwater flow direction or velocity needs to be determined, cluster piezometers or wells (i.e., wells completed to different depths in different boreholes at one data collection station) may be used. For groundwater quality monitoring or aquifer characteristic determination, monitoring wells or cluster wells should be used. In areas that are inaccessible to drill rigs (i.e., unstable surface soils), driven wells (drive points) may be used.

Siting of monitoring wells shall be performed after a preliminary estimation of groundwater flow direction. Typically, site visits, topographic mapping, regional/local hydrogeologic information, previously installed piezometers or monitoring wells, or information supplied by local drilling companies will provide information for siting wells. Flexibility should be maintained, so that well locations may be modified during the field investigation to account for site conditions (e.g., underground utilities). The elevation and horizontal location of all monitoring wells shall be determined through a site survey upon completion of well installation.

### 5.1 Well Installation

The methods discussed in this section are applicable to shallow, small diameter monitoring wells. Project-specific modifications to these methods shall be documented in the Sampling and Analysis Plan. These modifications may include larger diameter shallow wells, extraction wells, deep monitoring wells requiring surface casing and other specially constructed well types. Guidelines for monitoring well construction are given in Attachment A. Typical shallow monitoring well construction details are shown in Figures A-1 and A-2 in Attachment A for wells with flush-mounted and stick-up wells, respectively.

Note that these procedures discuss well installation using a PVC screen and riser pipe. Other materials such as stainless steel or Teflon also are available. Generally PVC is less expensive and easier to work with than either stainless steel or Teflon. A disadvantage to using PVC is the potential for degradation of the materials, or release (leaching) of constituents into the groundwater. Because of these concerns, justification for using PVC must be developed on a project-specific basis. The checklist shown in Attachment B provides a format for developing this justification.

Upon completion of each boring (refer to SOP F101 and F102 for Borehole and Sample Logging, and Soil and Rock Sample Acquisition, respectively), monitoring wells will usually be constructed using either 2-inch or 4-inch inside diameter (I.D.) screen and riser. Schedule 40 PVC, threaded, flush-joint casings with a

continuous #10 slot (0.010-inch), threaded, flush-joint PVC screen. A larger or smaller diameter screen may be used to accommodate site-specific geologic conditions. If wells are to be constructed over 100 feet in length, or in high traffic areas, or under other unusual conditions, Schedule 80 PVC may be used because of its greater strength.

An appropriate length of well screen shall be installed in each boring. The length of screen typically varies from 1 to 20 feet depending on site-specific conditions. For light nonaqueous phase liquid (LNAPL) applications, the screen should be installed such that at least 2 feet of screen is above the water table and the remainder of the screen extends below the water surface so that free product can enter the well. Should very shallow water table conditions be encountered, the screened interval in both the saturated and unsaturated zones may be reduced to ensure an adequate well seal above the screened interval. If this situation is expected, it should be addressed in the project plans, as necessary. A 6-inch section of PVC casing may be placed at the bottom of each screen to act as a settling cup for fines which may pass through the filter pack and screen.

Other applications may call for different screen placement depending on the zone to be monitored and the expected contaminants. For example, monitoring for dense non-aqueous phase liquids (DNAPLs) may require placing the screened interval in a "sump" at the base of the aquifer. Depending on the purpose of the monitoring well, the riser pipe may extend from the top of the screened interval to either 6 inches below the ground surface (for flush-mounted wells) to between approximately one and 2 feet above the ground surface for wells completed with stick-up.

The annular space around the screen is to be successively backfilled with a well graded quartz-sand, sodium bentonite and cement/bentonite grout as the hollow-stem augers are being withdrawn from the borehole. The sand size used in well construction will be appropriate for the formation monitored by the well. Sand shall carefully be placed from the bottom of the boring to a minimum of 2 feet (or 20 percent of the total screen length) above the top of the screened interval. A lesser distance above the top of the screened interval may be packed with sand if the well is very shallow to allow for placement of sealing materials.

A sodium bentonite seal at least two-foot thick shall be place above the sand pack. For deep wells, a bentonite slurry may be more appropriate than pellets due to problems with bridging in the annular space.

The annular space above the bentonite seal will be backfilled with a cement-bentonite grout consisting of 3 to 4 percent bentonite powder (by dry weight) or equivalent grout. The grout mixture shall be specified in the project plans. The grout will be tremied into the annular space greater than 20 feet high. If the annular space is less than 20 feet high, the grout may be poured directly into the annular space.

The depth intervals of all backfill materials shall be measured with a weighted measuring tape to the nearest 0.1 foot and recorded on the Field Well Construction Record (Attachment C) or in a field logbook.

## 5.2 Drive Points

Drive points may be constructed in one of two ways. If the drive point is hammered into place, no other well construction will take place. (Note that the well assembly is fabricated from 2-inch diameter stainless steel and includes a screen casing, and hardened point). The drive points will be sampled according to SOP F104, "Groundwater Sample Acquisition."

## 5.3 Surface Completion

There are several methods for surface completion of monitoring wells. Two such methods are discussed below.

The first method considers wells completed with stick-up. The aboveground section of the PVC riser pipe will be protected by installation of a 4- or 6-inch diameter, 5-foot long steel casing into the cement grout with locking cap and lock. The bottom of the surface casing will be placed at a minimum of 2-1/2, but not more than 3-1/2 feet below the ground surface. For very shallow wells, a steel casing of less than five-feet in length may be used, as space permits. The protective steel casing shall not fully penetrate the bentonite seal. A concrete apron shall be constructed around the steel casing.

The second method considers flush-mounted wells, typically installed where a stick-up installation would present a traffic hazard. The monitoring well shall be completed at the surface using a "flush" mount type cover. If the well is installed through a paved or concrete surface, the annular space shall be grouted to a depth of at least 2.5-feet and the well shall be finished with a concrete collar. If the well has not been installed through a paved or concrete surface, the well shall be completed by construction of a concrete apron. The concrete shall be crowned to meet the finished grade of the surrounding pavement, as required. If appropriate, the vault around the buried wellhead will have a water drain to the surrounding soil and a watertight cover.

Project specific tasks may require that all monitoring wells shall be labeled by metal stamping on the exterior of the protective steel casing or locking cap. A sign reading "Not For Potable Use or Disposal" also shall be firmly attached to each well. Alternately, well identification information may be stamped on a metal plate and attached to the well protective steel casing or embedded in the concrete apron, if appropriate.

## 5.4 Well Development

There are two stages of well development, initial and sampling. Sampling development is described in SOP F104, Groundwater Sample Acquisition. Initial development takes place after the completion materials have stabilized, as the last part of well construction.

The purposes of the initial development are to stabilize and increase the permeability of the filter pack around the well screen, to restore the permeability of the formation which may have been reduced by the drilling operations, and to remove fine-grained materials that may have entered the well or filter pack during installation. The selection of the well development method typically is based on drilling methods, well construction and installation details, and the characteristics of the formation. Any equipment that is introduced into the well during development shall be decontaminated in accordance with the SOP F501, entitled "Decontamination of Drilling Rigs and Monitoring Well Materials." A detailed discussion of well development is provided in Driscoll, 1986.

Well development shall not be initiated until a minimum of 24 hours has elapsed subsequent to well completion. This time period will allow the cement grout to set. Wells typically are developed using bailers, low-yield pumping, or surging with a surge block or air. The appropriate method shall be specified in the project plans.

In general, all wells shall be developed until well water runs relatively clear of fine-grained materials. Typical limits placed on well development may include any one of the following:

- Clarity of water based on visual determination.
- A minimum pumping time period (typically one hour for shallow wells 10 to 30 feet deep).
- A minimum borehole volume (typically three borehole volumes) or until well goes dry.
- Stability of specific conductance, turbidity, and temperature measurements (typically less than 10 percent change between three successive measurements).

In addition, a volume equal to any water added during drilling will be removed above and beyond the requirement specified above.

Well development limits shall be specified in project-specific plans. A record of the well development (Figure A-3 in Attachment A) also shall be completed to document the development process.

Usually, a minimum period of one week should elapse between the end of initial development and the first sampling event for a well. This equilibration period allows groundwater unaffected by the installation of the well to occupy the vicinity of the screened interval. However, this stabilization period may be adjusted based upon project-specific requirements.

## 5.5 Contaminated Materials Handling

SOP F504, entitled "Handling of Site Investigation Derived Waste," discusses the procedures to be used for the handling of auger cuttings, decontamination water, steam pad water, and development and purge water. Specific handling procedures should be delineated in the Sampling and Analysis Plan. In general, all site investigation generated wastes shall be containerized unless otherwise specified by the Sampling and Analysis Plan. The disposition of these wastes shall be determined after receipt of the appropriate analytical results.

## 5.6 Well Construction Records

Field Well Construction Records shall be completed by the Drilling Inspector for each monitoring well installed. These records preferably shall be completed as the well is being constructed. However, due to space limitations on this form it may be more practical to record well installation information in the field logbook and later transfer it to the Field Well Construction Record. If well construction information is recorded in the field logbook, it must be transferred to the appropriate form within 5 days, and prior to demobilization from the field.

Field Well Construction Records (in Attachment C), shall include not only well construction information, but also information pertaining to the amount of materials used for construction. Some of the following items shall be recorded on the Field Well Construction Record, or in the field logbook, as appropriate:

- Project name and location.
- Project number.

- Date and weather.
- Well identification designation.
- Drilling company and driller.
- Top of casing elevation (information collected after the site survey).
- Pay items including amount of screen and riser pipe used, amounts of cement, bentonite and sand used, and other well construction items.
- Well casing and borehole diameters.
- Elevations of (or depth to) top of steel casing, bottom of well, top of filter pack, top of bentonite seal, top of screen.

The information on the Field Well Construction Record will be used to generate a final Well Construction Record which combines the Field Boring and Well Construction Logs into one package. An example of all three documents is presented in Attachment C.

## 6.0 QUALITY ASSURANCE RECORDS

The Field Well Construction Record is the principle quality assurance record generated from well installation activities. Additionally, a Field Well Development Record shall also be completed, as well as pertinent comments in the field logbook.

## 7.0 REFERENCES

1. Driscoll, Fletcher, G., 1986, Groundwater and Wells, Johnson division. St. Paul, Minnesota. 2nd ed..

2. Roscoe Moss Company, 1990, Handbook of Ground Water Development. John Wiley & Sons. New York.

3. USEPA, September, 1986, RCRA Ground-Water Monitoring Technical Enforcement Guidance Document.

4. Aller, L. et al. , June 1989, Handbook of Suggested Practices for the Design and Installation of Ground Water Monitoring Wells. National Water Well Association. Dublin, Ohio.

ATTACHMENT A

# UST MONITORING WELL CONSTRUCTION AND FIELD OPERATIONS

## SPECIFICATIONS

Well permits required by state agencies are the responsibility of the contractor. All monitoring wells will be installed in accordance with the specifications set forth in the site-specific sampling and analysis plan. The wells will be constructed of either a 2-inch or 4-inch inside diameter (I.D.) flush joint threaded PVC well screen and riser casing depending on conditions encountered during borehole completion.

## DRILLING

During the drilling program, boreholes will be advanced using conventional hollow-stem auger drilling methods. If it is the opinion of the contractor that air or mud rotary drill methods are necessary, approval must be obtained from the Investigation Manager. Presentation of justification for a boring method change shall be presented prior to drilling.

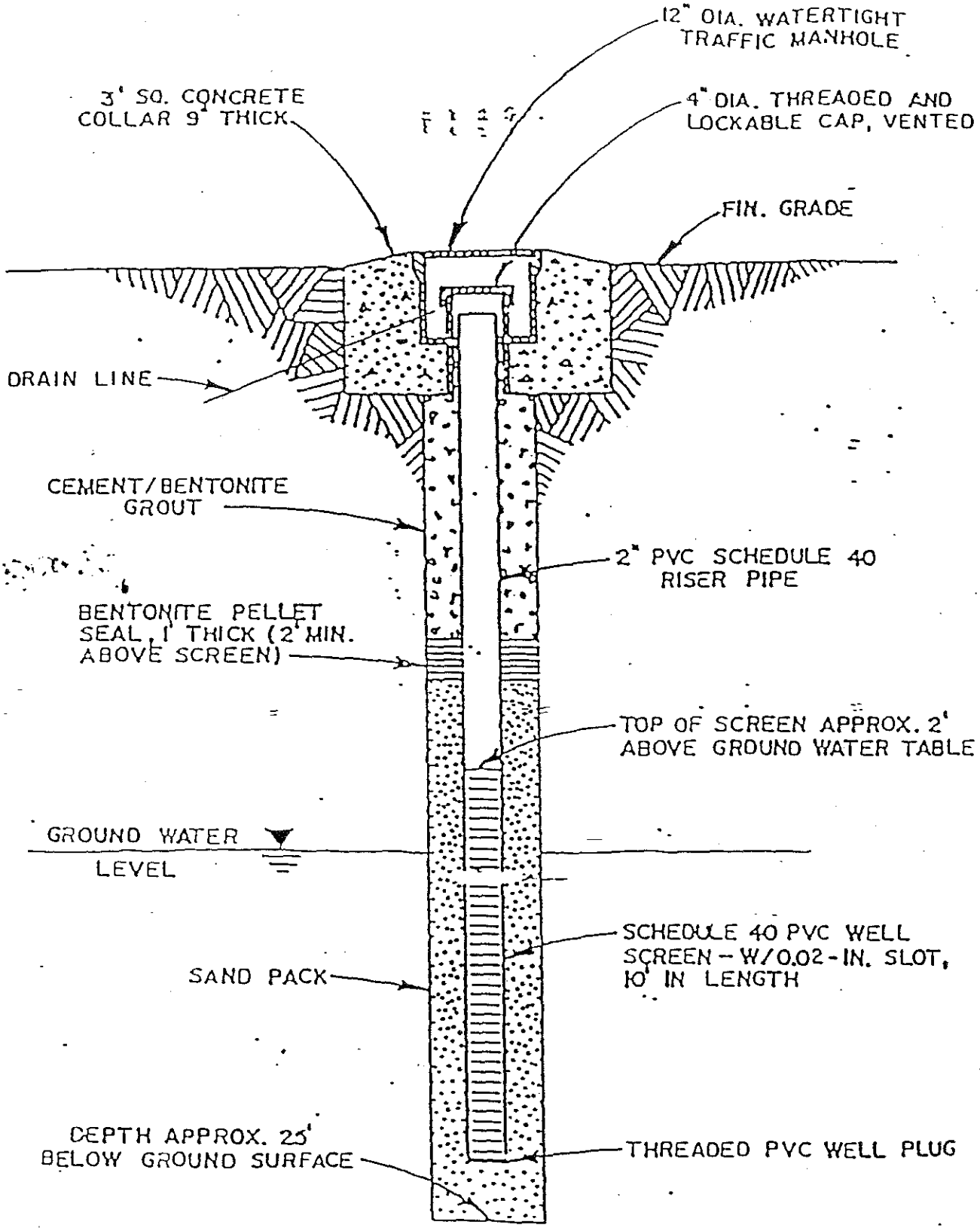
Well construction details are shown in Figures A-1 and A-2. A drill mounted on an All-Terrain-Vehicle (ATV) may be required for access to remote areas. Each rig will use necessary tools, supplies and equipment supplied by the contractor to drill each site. Drill crews should consist of an experienced driller and a driller assistant for work on each rig. A geologist, experienced in hazardous waste site investigations, shall be on site to monitor the drillers efforts and for air monitoring/safety control. Additional subcontractor personnel may be needed to transport water to the rigs, clean tools, assist in the installation of the security and marker pipes, construct the concrete aprons/collars and develop the wells. A potable water source in the plant will be designated by U.S. Steel.

Standard Penetration Tests (SPTs) will be performed in accordance with ASTM D-1586. Standard penetration tests will be performed at the following depths: 0.0-1.5 feet; 1.5-3.0 feet; 3.0-4.5 feet; and 5-foot centers thereafter. In cases where soil sampling for environmental analytical analysis is required, 24-inch spoon barrels may be used in the SPT to obtain a sufficient amount of sample for required analysis. A boring log of the soil type, stratification, consistency, and groundwater level will be prepared.

## SAMPLING

Soil samples of the subsurface materials will be collected at the interval specified in the Sample and Analysis Plan throughout the borehole in accordance with ASTM Method D-1586. Each soil sample will be screened in the field using an Hnu photoionizer, organic vapor detector or similar type direct readout instrument to identify the presence of volatile organic vapors within the soils. This field screening will provide a preliminary indication of the vertical and horizontal extent of contamination in order to select the optimum locations of other monitoring wells during the drilling program. Based on the field screening, two-inch or four-inch diameter monitoring wells will be installed at the locations where the most significant accumulation of contamination is encountered.

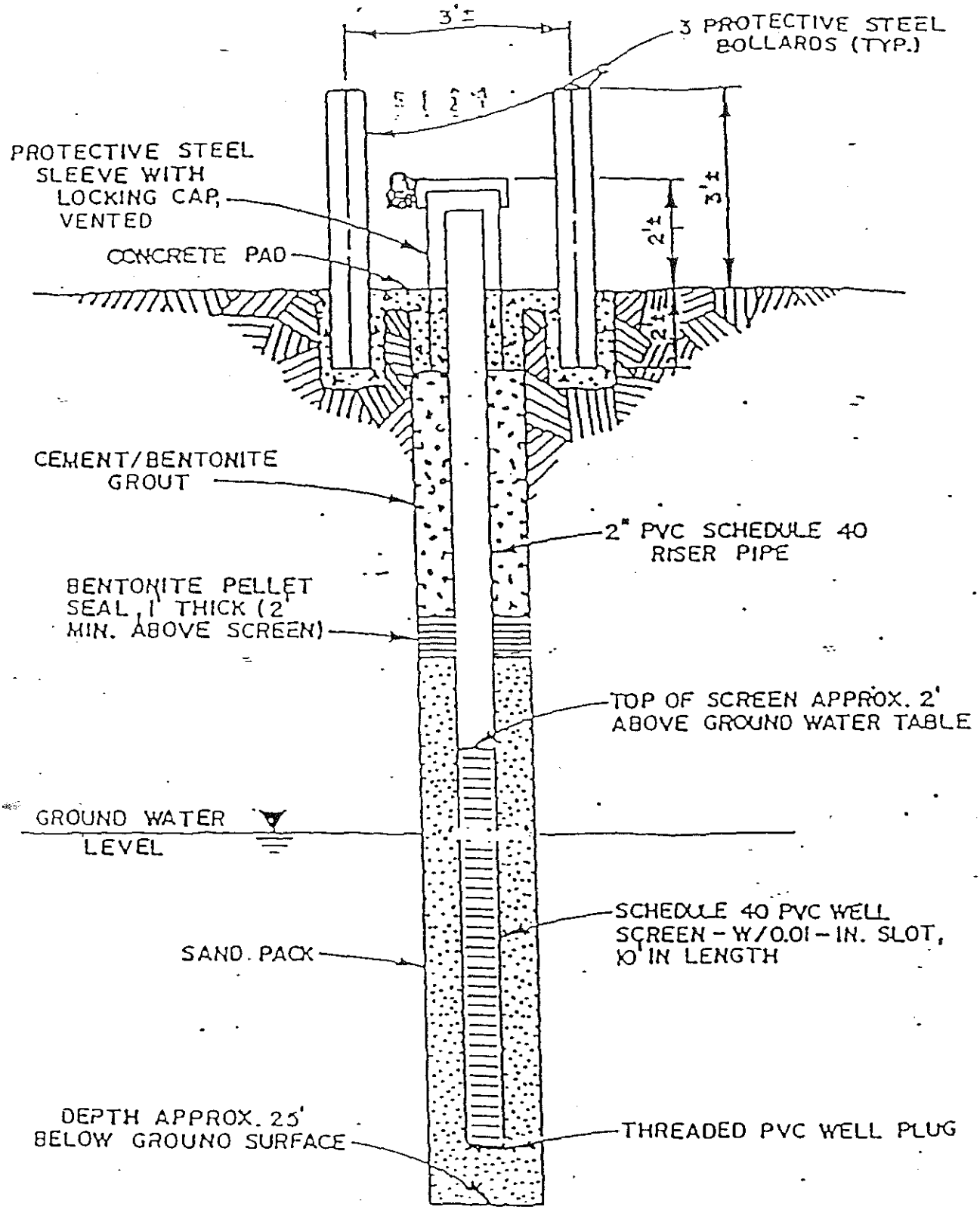
# FIGURE A-1




## MONITORING WELL CONSTRUCTION DETAIL (TRAFFIC AREA)

NOT TO SCALE

# FIGURE A-2




## MONITORING WELL CONSTRUCTION DETAIL
## (NON TRAFFIC AREA)

NOT TO SCALE

## WELL INSTALLATION

After completion of soil sampling and drilling to the specified depth, two-inch and/or four-inch (as required in the Sample and Analysis Plan) inside-diameter, flush-threaded Schedule 40 PVC (Schedule 80 in traffic areas) monitoring wells with slotted screens and well casings will be installed in the borehole. A 5- to 15-foot section of 0.01-inch slotted PVC well screen shall be used in each well. A sand pack will be placed around the slotted well screen extending to 2 feet above the top of the screen. A bentonite seal (minimum thickness of 1 foot) will be placed on top of the sand pack. Finally, a grout mixture of three to four percent bentonite powder (by dry weight) and seven gallons of water per 94 pound bag of cement, thoroughly mixed, will be placed in the borehole to insure a proper seal.

## WELL DEVELOPMENT

All wells will be developed not less than 24 hours following their installation to remove fine ground materials that may have entered the well during construction. Wells shall be developed until water runs relatively clear of fine-grained materials. Note that the water in some wells do not clear with continued development. Typical limits placed on well development may include any one of the following:

- Clarity of water based on visual determination.
- A maximum time period (typically one hour for shallow wells, well depth of 10 to 30 feet).
- A maximum well volume (typically three to five well volumes).
- Stability of specific conductance and temperature measurements (typically less than 10 percent change between three successive measurements).
- Clarity based on turbidity measurements (typically less than 50 NTU).

In addition, a volume equal to any water added during drilling will be removed above and beyond the requirement specified above.

Figure A-3 presents the Field Well Development Log used to document development data. This will be accomplished by either bailing or continuous, low-yield pumping. Equipment used for well installation that may have come in contact with potentially contaminated material will be decontaminated with a high pressure steam wash followed by a potable water rinse. SOPF5O4, entitled "Handling of Site Investigation Derived Waste", discusses the procedures to be used for the handling of auger cuttings, decontamination water, steam pad water, and development and purge water. Specific handling procedures should be delineated in the Sampling and Analysis Plan.

Supplies and equipment will be transported to the lay-down area designated by U.S. Steel. Any office space, trailers, etc., required for drilling, subsequent sampling and shipping shall be arranged and provided by the contractor.

## WELLHEAD COMPLETION

A four-inch diameter security pipe with a hinged locking cap will be installed over the well casing top and will be embedded approximately 2.5 feet into the grout.

There are two acceptable methods of completing the wellheads.

In traffic areas (and non-traffic areas where required), a "flush" mount type cover shall be built into a concrete pad as shown in Figure A-1. If the well is installed through a paved or concrete surface, the annular space between the casing and the borehole shall be grouted to a depth of at least 2.5 feet and finished with

a concrete collar. If the well is not installed through a concrete or paved medium and still finished as a high traffic area, a concrete apron measuring 5-foot by 5-foot by 0.5 foot will be constructed around each well. This apron/collar will be constructed of 3,000 psi ready-mixed concrete. The concrete will be crowned to provide and to meet the finished grade of surrounding pavement as required. The concrete pads can be constructed within five days after all of the wells have been installed.

In non-traffic areas the acceptable method of finishing a wellhead is shown in Figure A-2. Each well will be marked with three, Schedule 40 steel pipes, three-inch I.D., embedded in a minimum of 2.5-foot of 3,000 psi concrete. (The concrete used to secure the three pipes will be poured at the same time and be an integral part of the 5-foot by 5-foot by 0.5-foot concrete apron described above.) The security pipes will extend a minimum of 2.5 feet and maximum of 4.0 feet above the ground surface. The steel marker pipes will be filled with concrete and painted day-glo yellow or an equivalent. Attachment C presents Sample Field Test Boring Records and Field Well Construction Record Forms.

In all finished methods, the well covers will be properly labeled on the exterior of the security pipe locking cap and by labeling vertically on the exterior of the security pipe or manhole cover, as appropriate. The labeling shall consist of the identification specific to each well as described in the Sampling and Analysis Plan.

A sign reading "NOT FOR POTABLE USE OR DISPOSAL" shall be firmly attached to each well.

* The contractor or project team may supplement these requirements, but may not modify or delete them, in total or in part, without prior approval of the USEPA.

If any part of the above specifications is in conflict with the regulations set forth by the State, the State regulations take precedent.

# FIELD WELL DEVELOPMENT RECORD

Baker Environmental, Inc.

PROJECT: _____

CTO NO.: _____ WELL NO.: _____

DATE: _____

GEOLOGIST/ENGINEER: _____

| | DEVELOPMENT DATA | | | | | | |
|---|---|---|---|---|---|---|---|
| E START | TIME | CUMULATIVE VOLUME (gallons) | pH | TEMP (°C) | SPEC. COND. (µmhos/cm) | TEMP (°C) | COLOR AND TURBIDITY |
| E FINISH | | | | | | | |
| IAL WATER LEVEL (FT) | | | | | | | |
| AL WELL DEPTH (TD) | | | | | | | |
| L DIAMETER (INCHES) | | | | | | | |
| CULATED WELL VOLUME | | | | | | | |
| EHOLE DIAMETER (INCHES) | | | | | | | |
| EHOLE VOLUME | | | | | | | |
| UNT OF WATER ADDED NG DRILLING | | | | | | | |
| LOPMENT METHOD | | | | | | | |
| P TYPE | | | | | | | |
| L TIME (A) | | | | | | | |
| AGE FLOW (GPM)(B) | | | | | | | |
| L ESTIMATED ORAWAL AxB = | | | | | | | |
| VA READING | | | | | | | |

OBSERVATIONS/NOTES

# ATTACHMENT B

# ALTERNATE WELL CASING MATERIAL JUSTIFICATION

## ATTACHMENT B

## ALTERNATE WELL CASING MATERIAL JUSTIFICATION

The following is EPA's minimum seven point information requirements to justify the use of PVC as an alternate casing material for groundwater monitoring wells. If requested by EPA (USEPA Region IV), justification of the use of PVC should be developed by addressing each of the following items.

1. The Data Quality Objectives (DQOs) for the samples to be collected from wells with PVC casing as per EPA/540/G-87/003, "Data Quality Objectives for Remedial Response Activities."

2. The anticipated compounds and their concentration ranges.

3. The anticipated residence time of the sample in the well and the aquifer's productivity.

4. The reasons for not using other casing materials.

5. Literature on the adsorption characteristics of the compounds and elements of interest for the type of PVC to be used.

6. Whether the wall thickness of the PVC casing would require a larger annular space when compared to other well construction materials.

7. The type of PVC to be used and, if available, the manufacturers specifications, and an assurance that the PVC to be used does not leach, mask, react or otherwise interfere with the contaminants being monitored within the limits of the DQOs.

# ATTACHMENT C

## FIELD TEST BORING RECORD AND FIELD WELL CONSTRUCTION RECORD FORMS

# FIELD TEST BORING RECORD

PROJECT: Building P-64
S.O. NO.: 19010-51-SRV
BORING NO.: B-1
COORDINATES: EAST: NORTH:
ELEVATION: SURFACE: TOP OF STEEL CASING:

RIG: Mobile B-57

| | SPLIT SPOON | CASING | AUGERS | CORE BARREL | DATE | PROGRESS (FT) | WEATHER | WATER DEPTH (FT) | TIME |
|---|---|---|---|---|---|---|---|---|---|
| SIZE (DIAM.) | 1-3/8" ID | | 6-1/4" ID | | 5/31/91 | 14.0 | Sunny, 80°-90°F | — | — |
| LENGTH | 2.0' | | 5.0' | | | | | | |
| TYPE | STD. | | HSA | | | | | | |
| HAMMER WT. | 140 lb. | | | | | | | | |
| FALL | 30" | | | | | | — | | |
| STICK UP | | | | | | | — | — | |

REMARKS: Advanced boring to 14ft taking continuous 2-foot split-spoon samples; No monitoring Well installed - borehole grouted to surface

## DRILL RECORD / VISUAL DESCRIPTION

| O H | SOIL / ROCK | Sample ID / Type-No (N = No Samp) | Samp. Rec. (Ft & %) | SPT Blows Per 0.5' / RQD (Ft & %) | Lab Class / Pen. Rate | Lab MC % / PID (ppm) | Classification (Grain Size, Principal Constituents, Etc.) / Classification (Name, Grain Size, Principal Constituents, Etc.) | Color | Consist. or Density / Hardness | Moisture Content, Organic Content, Plasticity, and Other Observations / Weathering, Bedding, Fracturing, and Other Observations | SOIL / ROCK | ELEVATION |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | S-1 | 1.3/2.0 65% | 3 7 9 5 | | 0 | TOPSOIL, grass roots; | Tan gray | medium dense | dry | | 1.0' |
| 2 | 2.0 | | | | | | SAND, fine-grained, trace gravel, trace silt | Tan brown | Loose | moist to damp | | |
| 3 | | S-2 | 1.3/2.0 65% | 4 3 4 4 | | 0 | SAND, fine-grained, trace silt, trace grass roots | Tan brown | Loose | moist to damp | | |
| 4 | 4.0 | | | | | | | | | | | 4.0' |
| 5 | | S-3 | 2.0/2.0 100% | 11 12 10 8 | | 0 | SAND, fine to medium-grained, trace silt | Tan brown orange | Medium dense | moist to wet; water table noted at 6.0' | | |
| 6 | 6.0 | | | | | | | | | | | |
| 7 | | S-4 | 1.8/2.0 90% | 3 4 3 4 | | 0 | | | | | | 7.0' |
| 8 | 8.0 | | | | | | SAND, medium to coarse-grained, trace silt | Tan gray orange | Loose | wet | | |
| 9 | | S-5 | 2.0/2.0 100% | 1 0 1 1 | | 0 | | | | | | 9.0' |
| 10 | 10.0 | | | | | | SAND, medium-grained, trace silt | Gray orange | Loose | wet | | |

DRILLING CO.: AIEC Associates BAKER REP.: R. Bovelli
DRILLER: M. Miller BORING NO.: B-1 SHEET 1 OF 2

# FIELD TEST BORING RECORD

PROJECT: Building P-64
S.O. NO.: 19010-51-SRN BORING NO.: B-1

| DEPTH | SOIL / ROCK | Sample ID / Type-No. (N = No Samp.) | Samp. Rec. (Ft. & %) | SPT Blows Per 0.5' / RQD (Ft. & %) | Lab Class / Pen. Rate | Lab. M.C. % / PID (ppm) | Classification (Grain Size, Principal Constituents, Etc.) / Classification (Name, Grain Size, Principal Constituents, Etc.) | Color | Consist or Density / Hardness | Moisture Content, Organic Content, Plasticity, and Other Observations / Weathering, Bedding, Fracturing, and Other Observations | SOIL / ROCK | ELEVATION |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | 2.0 / 2.0 | 1 | | | SAND, medium-grained, trace silt | Gray | very loose | wet | | |
| 2 | 12.0 | S-6 | 100% | 1 | | 0 | | | | | | |
| 3 | | S-7 | 2.0 / 2.0 | 0 | | | SAND, medium-grained, trace silt | Gray | very loose | wet | | |
| 4 | 14.0 | | 100% | 1 | | 0 | | | | | | 14.0' |
| 5 | | | | | | | End of boring at 14.0 ft. | | | | | |
| 6 | | | | | | | | | | | | |
| 7 | | | | | | | | | | | | |
| 8 | | | | | | | | | | | | |
| 9 | | | | | | | | | | | | |
| 10 | | | | | | | | | | | | |
| 11 | | | | | | | | | | | | |
| 12 | | | | | | | | | | | | |
| 13 | | | | | | | | | | | | |
| 14 | | | | | | | | | | | | |
| 15 | | | | | | | | | | | | |
| 16 | | | | | | | | | | | | |
| 17 | | | | | | | | | | | | |
| 18 | | | | | | | | | | | | |
| 19 | | | | | | | | | | | | |
| 20 | | | | | | | | | | | | |

DRILLING CO.: ATEC Associates BAKER REP.: R. Bovelli
DRILLER: M. Miller BORING NO.: B-1 SHEET 2 OF 2

# TEST BORING RECORD

Baker Environmental, Inc.

PROJECT: Building P-64
S.O. NO.: 19010-51-SRN
COORDINATES: EAST: _____ NORTH: _____
ELEVATION: SURFACE: _____ TOP OF PVC CASING: _____
BORING NO.: B-1

RIG: Mobile B-57

| | SPLIT SPOON | CASING | AUGERS | CORE BARREL | DATE | PROGRESS (FT) | WEATHER | WATER DEPTH (FT) | TIME |
|---|---|---|---|---|---|---|---|---|---|
| SIZE (DIAM.) | 1-3/8"ID | | 6-1/4" ID | | 5/31/91 | 14.0 | Sunny, 80°-90°F | ------ | ---- |
| LENGTH | 2.0' | | 5.0' | | | | | | |
| TYPE | STD. | | HSA | | | | | | |
| HAMMER WT. | 140# | | | | | | | | |
| FALL | 30" | | | | | | | | |
| STICK UP | | | | | | | | | |

REMARKS: Advanced boring to 14 ft. taking continuous 2-foot split-spoon samples; no monitoring well installed - borehole grouted to surface.

SAMPLE TYPE
S = Split Spoon, A = Auger, T = Shelby Tube, W = Wash, = Air Rotary, C = Core, = Denison, P = Piston, N = No Sample

DEFINITIONS
SPT = Standard Penetration Test (ASTM D-1586) (Blows/0.5')
RQD = Rock Quality Designation (%)
Lab Class. = USCS (ASTM D-2487) or AASHTO (ASTM D-3282)
Lab Moist. = Moisture Content (ASTM D-2216) Dry Weight Basis

| Depth (Ft.) | Sample Type and No. | Samp. Rec. Ft. & % | SPT or RQD | Lab. Class. or Pen. Rate | PID (ppm) | Visual Description | Elevation |
|---|---|---|---|---|---|---|---|
| 0 | | | 3 | | | TOPSOIL, grass roots; tan, gray; medium dense; dry | |
| 1 | S-1 | 1.3/2.0 65% | 7 9 5 | | 0 | 1.0' | |
| 2 2.0 | | | | | | SAND, fine-grained, trace gravel, trace silt; tan, brown; loose; moist to damp | |
| 3 | S-2 | 1.3/2.0 65% | 4 3 4 4 | | 0 | SAND, fine-grained, trace silt, trace grass roots; tan, brown; loose; moist to damp | |
| 4 4.0 | | | | | | 4.0' | |
| 5 | S-3 | 2.0/2.0 100% | 11 12 10 8 | | 0 | SAND, fine to medium-grained, trace silt; tan, brown, orange; medium dense; moist to wet; water table at 6.0' | |
| 6 6.0 | | | | | | 7.0' | |
| 7 | S-4 | 1.8/2.0 90% | 3 4 3 4 | | 0 | SAND, medium to coarse-grained, trace silt; tan, gray, orange; loose; wet | |
| 8 8.0 | | | | | | 9.0' | |
| 9 | S-5 | 2.0/2.0 100% | 1 0 1 1 | | 0 | SAND, medium-grained, trace silt; gray, orange; very loose; wet | |
| 10 0.0 | | | | | | Match to Sheet 2 | |

DRILLING CO.: ATEC Associates BAKER REP.: R. Bonelli

# TEST BORING RECORD

Baker Environmental, Inc.

PROJECT: Building P-64
S.O. NO.: 19010-51-SRN BORING NO.: B-1

**SAMPLE TYPE**
S = Split Spoon A = Auger
T = Shelby Tube W = Wash
R = Air Rotary C = Core
D = Denison P = Piston
N = No Sample

**DEFINITIONS**
SPT = Standard Penetration Test (ASTM D-1586) (Blows/0.5')
RQD = Rock Quality Designation (%)
Lab Class. = USCS (ASTM D-2487) or AASHTO (ASTM D-3282)
Lab Moist. = Moisture Content (ASTM D-2216) Dry Weight Basis

| Depth (Ft.) | Sample Type and No. | Samp. Rec. (Ft. & %) | SPT or RQD | Lab. Class. or Pen. Rate | PID (ppm) | Visual Description | Elevation |
|---|---|---|---|---|---|---|---|
| 11 | S-6 | 2.0 / 2.0 100% | 1 1 0 1 | | 0 | SAND, medium-grained, trace silt; gray; very loose; wet | |
| 12.0 | | | | | | | |
| 13 | S-7 | 2.0 / 2.0 100% | 0 0 1 1 | | 0 | SAND, medium-grained, trace silt; gray; very loose; wet | |
| 14.0 | | | | | | 14.0' | |
| 14 | | | | | | End of Boring at 14.0' | |
| 15 | | | | | | | |
| 19 | | | | | | | |
| 20 | | | | | | | |
| 21 | | | | | | | |
| 22 | | | | | | | |
| 23 | | | | | | | |
| 24 | | | | | | | |
| 25 | | | | | | | |
| 26 | | | | | | | |
| 27 | | | | | | | |
| 28 | | | | | | | |
| 30 | | | | | | | |

DRILLING CO.: ATEC Associates BAKER REP.: R. Bonelli
DRILLER: M. Miller BORING NO.: B-1 SHEET 2 OF 2

# FIELD TEST BORING RECORD

Baker Environmental, Inc

PROJECT: Building P-64
CTO NO.: 19010-SY-SBRN
COORDINATES: EAST: _____ NORTH: _____
ELEVATION: SURFACE: 13.94 TOP OF STEEL CASING: 13.66
BORING NO.: MW-1

RIG: Mobile B-57

| | SPLIT SPOON | CASING | AUGERS | CORE BARREL | DATE | PROGRESS (FT) | WEATHER | WATER DEPTH (FT) | TIME |
|---|---|---|---|---|---|---|---|---|---|
| SIZE (DIAM.) | 1-3/8" ID | | 6-1/4" ID | | 5/29/91 | 14.0 | Sunny, 70°-80°F | -- | -- |
| LENGTH | 2' | | 5' | | 5/30/91 | -- | Sunny, 80°-90°F | 6.64 | 24 hrs |
| TYPE | STD | | HSA | | | | | | |
| HAMMER WT. | 140 lb. | | | | | | | | |
| FALL | 30" | | | | | | | | |
| STICK UP | | | | | | | | | |

REMARKS: Advanced boring to 14ft, taking continuous 2-foot split-spoon samples; Monitoring well installed at 14.35 ft

## DRILL RECORD / VISUAL DESCRIPTION

| DEPTH | SOIL / ROCK | Sample ID / Type-No. (N = No Samp.) | Samp. Rec. (Ft. & %) | SPT Blows Per 0.5' / RQD (Ft. & %) | Lab. Class / Pen. Rate | Lab. M.C. % / PID (ppm) | Classification (Grain Size, Principal Constituents, Etc.) / Classification (Name, Grain Size, Principal Constituents, Etc.) | Color | Consist. or Density / Hardness | Moisture Content, Organic Content, Plasticity, and Other Observations / Weathering, Bedding, Fracturing, and Other Observations | SOIL / ROCK | ELEVATION |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | S-1 | 1.2/2.0 60% | 16 12 6 5 | | 0 | SAND, fill material, trace gravel | brown-gray | medium dense | dry | | |
| 2 | 2.0 | | | | | | SAND, fill material, trace carbon trace glass | brown-gray-black | loose | dry | | |
| 3 | | S-2 | 1.4/2.0 70% | 4 4 4 3 | | 0 | SAND, fine grained, trace gravel, trace silt | gray-brown | very loose | damp to moist | 3.0' | 10.9 |
| 4 | 4.0 | | | | | | | | | | 4.0' | 9.9 |
| 5 | | S-3 | 1.8/2.0 90% | 1 1 1 1 | | 0 | SAND, fine to medium-grained, trace silt, trace clay | gray-brown | very loose | damp to moist; water-table noted at 5.5-6.0' | | |
| 6 | 6.0 | | | | | | SAND, fine to medium-grained, trace silt | gray-white | loose | wet; petroleum odor noted in spoon sample | | |
| 7 | | S-4 | 2.0/2.0 100% | 1 2 3 5 | | 1-3 | | | | | 7.0' | 7.3 |
| 8 | 8.0 | | | | | | SAND, medium to coarse-grained, trace silt, | gray-white-brown | loose | wet; petroleum odor noted in spoon sample | | 6.9 |
| 9 | | S-5 | 0.95/2.0 48% | 3 1 1 1 | | 0.5 | | | | | | |
| | 10.0 | | | | | | | | | | | |

DRILLING CO.: ATEC Associates BAKER REP.: R. Powell
DRILLER: W. Miller BORING NO.: MW-1

# FIELD TEST BORING RECORD

PROJECT: Building P-64
CTO NO.: 19010-51-SPN
BORING NO.: MW-1

| DEPTH | SOIL / ROCK | Sample ID — Type-No. (N = No Samp.) | Samp. Rec. (Ft. & %) | SPT Blows Per 0.5' / RQD (Ft. & %) | Lab. Class / Pen. Rate | Lab. M.C. % (ppm) | Classification (Grain Size, Principal Constituents, Etc.) / Classification (Name, Grain Size, Principal Constituents, Etc.) | Color | Consistency or Density / Hardness | Moisture Content, Organic Content, Plasticity, and Other Observations / Weathering, Bedding, Fracturing, and Other Observations | SOIL / ROCK | ELEVATION |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | 20/20 | 2 | | 0 | SAND, five to medium-grained, trace silt | dark gray | very loose | wet; petroleum odor noted in spoon sample | | |
| 2 | 2.0 | S-6 | 100% | 1 | | | | | | | | |
| 3 | | | 20/20 | 1 | | 0 | SAND, five to medium-grained, trace silt | dark gray gray-brown | very loose | wet; petroleum odor noted in spoon sample | | |
| 4 | 14.0 | S-7 | 100% | 1 | | | | | | 14.0' | | -0. |
| 5 | | | | | | | End of boring at 14.0' | | | | | |
| 6 | | | | | | | | | | | | |
| 8 | | | | | | | | | | | | |
| 9 | | | | | | | | | | | | |
| 0 | | | | | | | | | | | | |
| 1 | | | | | | | | | | | | |
| 2 | | | | | | | | | | | | |
| 3 | | | | | | | | | | | | |
| 4 | | | | | | | | | | | | |
| 5 | | | | | | | | | | | | |
| 6 | | | | | | | | | | | | |
| 7 | | | | | | | | | | | | |
| 8 | | | | | | | | | | | | |
| 9 | | | | | | | | | | | | |

DRILLING CO.: ATEC Associates
BAKER REP.: R Bonelli

# FIELD WELL CONSTRUCTION LOG

Baker Environmental, Inc.

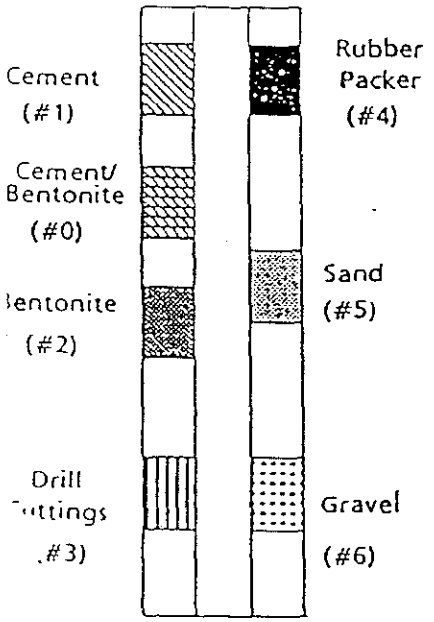
PROJECT: Building P-64 DATE: 5/29/91
CTO NO.: 19010-SI-SRLV BORING NO.: MW-1
COORDINATES: EAST: NORTH:
ELEVATION: SURFACE: 13.94 TOP OF ~~STEEL~~ PVC CASING: 13.66

## Pay Items

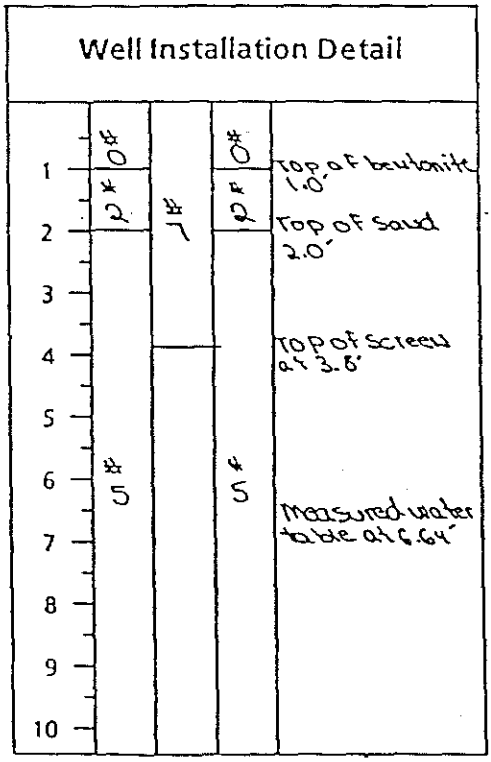
| Item | Quantity | Unit | Remarks |
|---|---|---|---|
| Sand: Industrial #2 | 3½ | bags | |
| Bentonite - Pellets | ½ | bucket | |
| Screen: #001 Slot 2-inch PVC | 10 | LF | |
| Casing - 2in PVC | 4 | LF | |
| Manhole Cover | 1 | ea | |
| Locking Cap + lock (#2006) | 1 | ea | |
| Cement / bentonite | 1 | LF | |

| WELL INFORMATION | DIAM. (INCHES) | TYPE | TOP DEPTH (FT.) | BOTTOM DEPTH (FT.) |
|---|---|---|---|---|
| Well Casing | 2.0 | Sch 40 PVC | 0.3 | 3.8 |
| Well Screen | 2.0 | Sch 40 PVC | 3.8 | 13.9 |

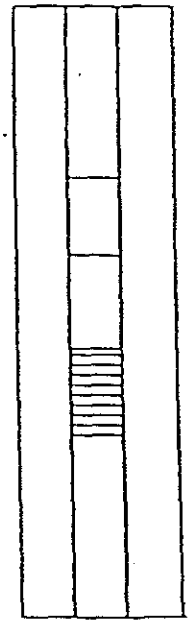
Backfill Key




Well Installation Detail




Well Key




DRILLING CO.: AFC Associates BAKER REP.: R Boxell.
DRILLER: M. Miller BORING NO.: MW-1 SHEET 1 OF 2

# TEST BORING AND WELL CONSTRUCTION RECORD

PROJECT: Building P-64
S.O. NO.: 19010-51-SRN
COORDINATES: EAST:
ELEVATION: SURFACE: 13.94
BORING NO.: MW-1
NORTH:
TOP OF PVC CASING: 13.66

RIG: Mobile B-57

| | SPLIT SPOON | CASING | AUGERS | CORE BARREL | DATE | PROGRESS (FT) | WEATHER | WATER DEPTH (FT) | TIME |
|---|---|---|---|---|---|---|---|---|---|
| SIZE (DIAM.) | 1-3/8" ID | | 6-1/4" ID | | 5/29/91 | 14.0 | sunny, 70°-80° F | --- | --- |
| LENGTH | 2.0' | | 5.0' | | 5/30/91 | --- | sunny, 80°-90° F | 6.64 | 24 hrs. |
| TYPE | STD | | HSA | | | | | | |
| HAMMER WT. | 140 lb. | | | | | | | | |
| FALL | 30" | | | | | | | | |
| STICK UP | | | | | | | | | |

REMARKS: Advanced boring to 14 ft. taking continuous 2-foot split-spoon samples; monitoring well installed at 14.35 ft.

SAMPLE TYPE
S = Split Spoon A = Auger
T = Shelby Tube W = Wash
R = Air Rotary C = Core
D = Denison P = Piston
N = No Sample

| WELL INFORMATION | DIAM | TYPE | TOP DEPTH (FT) | BOTTOM DEPTH (FT) |
|---|---|---|---|---|
| Well Casing | 2" | Sch. 40 PVC, flush-jointed | 0.28 | 3.8 |
| Well Screen | 2" | Sch. 40 PVC, flush-jointed | 3.8 | 13.9 |

| Depth (Ft.) | Sample Type and No | Samp. Rec. Ft. & % | SPT or RQD | Lab. Class. or Pen. Rate | PID (ppm) | Visual Description | Well Installation Detail | Elevation Ft. MSL |
|---|---|---|---|---|---|---|---|---|
| 2.0 | S-1 | 1.2 / 2.0 60% | 16 12 6 5 | | 0 | SAND, fill material, trace gravel; brown-gray; medium dense; dry | Top of bentonite at 1.0' | |
| | | | | | | SAND, fill material, trace carbon frags; brown-gray-black; loose; dry | Top of sand at 2.0' | |
| 4.0 | S-2 | 1.4 / 2.0 70% | 4 4 4 3 | | 0 | 3.0' SAND, fine-grained, trace gravel, trace silt; gray-brown; very loose; damp to moist | Top of screen at 3.8' | 10.94' |
| 6.0 | S-3 | 1.8 / 2.0 90% | 1 1 1 1 | | 0 | 4.0' SAND, fine to medium - grained, trace silt, trace clay; gray-brown; very loose; damp to moist; water table at 5.5' - 6.0' | | 9.94' |
| 8.0 | S-4 | 2.0 / 2.0 100% | 1 2 3 5 | | 1-3 | 7.0' SAND, fine to medium - grained, trace silt; gray-white; loose; wet; petroleum odor noted in spoon sample | Measured water table at 6.64' | 7.30' |
| 10.0 | S-5 | 9S / 2.0 48% | 3 1 1 1 | | 0.5 | SAND, medium to coarse-grained, trace silt; gray-white-brown; loose; wet; petroleum odor noted in spoon sample | | 6.94' |
| | | | | | | Match to Sheet 2 | | |

DRILLING CO.: ATEC Associates
DRILLER: M. Miller
BAKER REP.: R. Bonelli
BORING NO.: MW-1

# F104
# *GROUNDWATER SAMPLE ACQUISITION*

# GROUNDWATER SAMPLE ACQUISITION

## 1.0 PURPOSE

The purpose of this guideline is to provide general reference information on the sampling of groundwater wells. The methods and equipment described are for the collection of water samples from the saturated zone of the subsurface.

## 2.0 SCOPE

This guideline provides information on proper sampling equipment and techniques for groundwater sampling. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described should be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methods.

## 3.0 DEFINITIONS

None.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other, approved procedures are developed. The Project Manager is responsible for development of documentation of procedures which deviate from those presented herein.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the specific groundwater sampling techniques and equipment to be used, and documenting these in accordance with the Sampling and Analysis Plan. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and that personnel performing sampling activities have been briefed and trained to execute these procedures.

Sampling Personnel - It is the responsibility of the field sampling personnel to follow these procedures, or to follow documented, project-specific procedures as directed by the Field Team Leader and the Project Manager. The sampling personnel are responsible for the proper acquisition of groundwater samples.

## 5.0 PROCEDURES

To be useful and accurate, a groundwater sample must be representative of the particular zone being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of testing in order to minimize any changes in water quality parameters.

The groundwater sampling program should be developed with reference to ASTM D4448-85A, Standard Guide for Sampling Groundwater Monitoring Wells (Attachment A). This reference is not intended as a monitoring plan or procedure for a specific application, but rather is a review of methods. Specific methods shall be stated in the Sampling and Analysis Plan (SAP).

Methods for withdrawing samples from completed wells include the use of pumps, compressed air, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of the groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water due to sampling techniques. In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with the groundwater due to normal flow patterns, but the well water above the screened section will remain largely isolated and become stagnant. To safeguard against collecting non-representative stagnant water in a sample, the following approach should be followed during sample withdrawal:

1. All monitoring wells shall be pumped or bailed prior to withdrawing a sample. Evacuation of three to five volumes is recommended for a representative sample.

2. Wells that can be pumped or bailed to dryness with the sampling equipment being used, shall be evacuated and allowed to recover prior to sample withdrawal. If the recovery rate is fairly rapid and time allows, evacuation of at least three well volumes of water is preferred; otherwise, a sample will be taken when enough water is available to fill the sample containers.

Stratification of contaminants may exist in the aquifer formation. This is from concentration gradients due to dispersion and diffusion processes in a homogeneous layer, and from separation of flow streams by physical division (for example, around clay lenses) or by contrasts in permeability (for example, between a layer of silty, fine sand and a layer of medium sand).

Purging rates and volumes for non-production wells during sampling development should be moderate; pumping rates for production wells should be maintained at the rate normal for that well. Excessive pumping can dilute or increase the contaminant concentrations in the recovered sample compared to what is representative of the integrated water column at that point, thus result in the collection of a non-representative sample. Water produced during purging shall be collected, stored or treated and discharged as allowed. Disposition of purge water is usually site-specific and must be addressed in the Sampling and Analysis Plan.

## 5.1 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform with EPA regulations for the appropriate contaminants and to the specific Quality Assurance Project Plan.

The following list is an example of the type of equipment that generally must be on hand when sampling groundwater wells:

1. Sample packaging and shipping equipment: Coolers for sample shipping and cooling, chemical preservatives, and appropriate packing cartons and filler, labels and chain-of-custody documents.

2. Field tools and instrumentation: PID; thermometer; pH meter; turbidity meter; specific conductivity meter; appropriate keys (for locked wells) or bolt-cutter; tape measure; plastic sheeting; water-level indicator; calibrated buckets and, where applicable, flow meter.

3. Pumps

   a. Shallow-well pumps: Centrifugal, Packer Pumps, pitcher, suction, or peristaltic pumps with droplines, air-lift apparatus (compressor and tubing), as applicable.

   b. Deep-well pumps: Submersible pump and electrical power generating unit, bladder pump with compressed air source, or air-lift apparatus, as applicable.

4. Tubing: Sample tubing such as teflon, polyethylene, polypropylene, or PVC. Tubing type shall be selected based on specific site requirements and must be chemically inert to the groundwater being sampled.

5. Other Sampling Equipment: Bailers, teflon-coated wire, stainless steel single strand wire, and polypropylene monofilament line (not acceptable in EPA Region I) with tripod-pulley assembly (if necessary).

6. Pails: Plastic, graduated.

7. Decontamination equipment and materials: discussed in SOP F501 and F502.

Ideally, sample withdrawal equipment should be completely inert, economical, easily cleaned, sterilized, and reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

## 5.2 Calculations of Well Volume for Purging

To insure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to determine the volume of standing water in the well pipe or casing. The volume can be easily calculated by the following method. Calculations shall be entered in the field logbook:

1. Obtain all available information on well construction (location, casing, screens, etc.).

2. Determine inside diameter of well or casing (D).

3. Measure and record static water level (DW-depth to water below ground level or top of casing reference point) to the nearest 0.01-foot, using one of the methods described in Section 5.1 of SOP F202.

4. Determine the depth of the well (TD) to the nearest 0.01-foot by sounding using a clean, decontaminated weighted tape measure, referenced to the top of PVC casing or ground surface.

5. Calculate the volume of water in the casing:

$$V_W = \frac{\pi D_2}{4} (TD - DW)$$

$$V_{gal} = V_W \times 7.48 \text{ gallons/ft}^3$$

Where:

$V_w$=Volume of water standing in well in cubic feet (i.e., one well volume)
$\pi$=pi, 3.14
D=Inside diameter of well, in feet
TD=Total depth of well in feet (below ground surface or top of casing)
DW=Depth to water in feet (below ground surface or top of casing)

6. Calculate the minimum number of gallons to be evacuated before sampling. (Note: $V_{purge}$ should be rounded to the next highest whole gallon. For example, 7.2 gallons should be rounded to 8 gallons.)

$$V_{purge} = Vgal\ (\#\ Well\ Vol)$$

Where:

$V_{gal}$ = Volume of water in well in gallons
$V_{purge}$ = Volume of water to be purged from well in gallons
# Well Vol. = Number of well volumes of water to be purged from the well (typically three to five)

Table 5-1 lists gallons and cubic feet of water per standing foot of water for a variety of well diameters.

TABLE 5-1
WELL VOLUMES

| Diameter of Casing or Hole (in.) | Gallons per Foot of Depth | Cubic Feet per Foot of Depth |
|---|---|---|
| 1 | 0.041 | 0.0055 |
| 2 | 0.163 | 0.0218 |
| 4 | 0.653 | 0.0873 |
| 6 | 1.469 | 0.1963 |
| 8 | 2.611 | 0.3491 |
| 10 | 4.080 | 0.5454 |

## 5.3 Evacuation of Static Water (Purging)

The amount of purging a well should receive prior to sample collection will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of that aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer.

For defining a contaminant plume, a representative sample of only a small volume of the aquifer is required. These circumstances require that the well be pumped enough to remove the stagnant water but not enough to induce significant groundwater flow from a wide area. Generally, three to five well volumes are considered effective for purging a well.

An alternative method of purging a well, and one accepted in EPA Regions I and IV, is to purge a well continuously (usually using a low volume, low flow pump) while monitoring specific conductance, pH, turbidity, and water temperature until the values stabilize. Values are considered to have stabilized when deviation is less than 10 percent of the mean. The well is considered properly purged when the values have stabilized.

If a well is dewatered before the required volume is purged, the sample should be collected from the well once as a sufficient volume of water has entered the well. In order to avoid stagnation, the well should not be allowed to fully recharge before the sample is collected. The field parameters (pH, conductance, and temperature) should be recorded when the well was dewatered.

The Project Manager shall define the objectives of the groundwater sampling program in the Sampling and Analysis Plan, and provide appropriate criteria and guidance to the sampling personnel on the proper methods and volumes of well purging.

### 5.3.1 Evacuation Devices

The following discussion is limited to those devices which are commonly used at hazardous waste sites. Note that all of these techniques involve equipment which is portable and readily available.

Bailers - Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of pipe with a sealed bottom (bucket-type bailer) or, as is more useful and favored, with a ball check-valve at the bottom. An inert line (e.g., Teflon-coated) is used to lower the bailer and retrieve the sample.

Advantages of bailers include:

- Few limitations on size and materials used for bailers.
- No external power source needed.
- Inexpensive.
- Minimal outgassing of volatile organics while the sample is in the bailer.
- Relatively easy to decontaminate and use.

Limitations on the use of bailers include the following:

- Limited volume of sample.
- Time consuming to remove stagnant water using a bailer.
- Collection and transfer of sample may cause aeration.
- Use of bailers is physically demanding, especially in warm temperatures at protection levels above Level D.
- Unable to collect depth-discrete sample.

Suction Pumps - There are many different types of inexpensive suction pumps including centrifugal, diaphragm, peristaltic, and pitcher pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low volume pump (generally not suitable for well purging) that uses rollers to squeeze a flexible tubing, thereby creating suction. This tubing can be dedicated to a well to prevent cross contamination. The pitcher pump is a common farm hand-pump.

Advantages of suction pumps include:

- Few limitations with regards to well diameter
- Inexpensive
- Portable
- Readily available
- Tubing can be dedicated or easily decontaminated

Limitations on the use of suction pumps include the following:

- External power source
- Vacuum will cause loss of dissolved gas, including volatile organics
- Restricted to areas with water levels within 10 to 25 feet of the ground surface
- Internal components of the pumps may be difficult to decontaminate

Gas-Lift Samples - This group of samplers uses gas pressure either in the annulus of the well or in a venturi to force the water up a sampling tube. These pumps are also relatively inexpensive. Gas lift pumps are more suitable for well development than for sampling because the samples may be aerated, leading to pH changes and subsequent trace metal precipitation or loss of volatile organics. An inert gas such as nitrogen is generally used as a gas source.

Submersible Pumps - Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed air or electricity. The operation principles vary and the displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for two-inch diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include:

- Potentially low delivery rates.
- Many models of these pumps are expensive.
- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding the impellers with some of these pumps.
- Decontamination of internal components is difficult and time-consuming.

## 5.4 Sampling

The sampling approach consisting of the following, should be developed as part of the Sampling and Analysis Plan prior to the field work:

1. Background and objectives of sampling.
2. Brief description of area and waste characterization.
3. Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).
4. Sampling equipment to be used.
5. Intended number, sequence volumes, and types of samples. If the relative degrees of contamination between wells is unknown or insignificant, a sampling sequence which facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these should be sampled last to reduce the risk of cross-contamination between wells as a result of the sampling procedures.
6. Sample preservation requirements.
7. Schedule.
8. List of team members.
9. Other information, such as the necessity for a warrant or permission of entry, requirement for split samples, access problems, location of keys, etc.

### 5.4.1 Sampling Methods

The collection of a groundwater sample includes the following steps:

1. First open the well cap and use volatile organic detection equipment (HNu or OVA) on the escaping gases at the well head to determine the need for respiratory protection. This task is usually performed by the Field Team Leader, Health and Safety Officer, or other designee.

2. When proper respiratory protection has been donned, measure the total depth and water level (with decontaminated equipment) and record these data in the field logbook. Calculate the fluid volume in the well according to Section 5.2 of this SOP.

3. Lower purging equipment or intake into the well to a distance just below the water level and begin water removal. Collect the purged water and dispose of it in an acceptable manner (e.g., DOT-approved 55-gallon drum).

4. Measure the rate of discharge frequently. A bucket and stopwatch are most commonly used; other techniques include using pipe trajectory methods, weir boxes or flow meters. Record the method of discharge measurement.

5. Observe peristaltic pump intake for degassing "bubbles" and all pump discharge lines. If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.

6. Purge a minimum of three to five well volumes before sampling. In low permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recharge as necessary, but preferably to 70 percent of the static water level, and then sample.

7. Record measurements of specific conductance, temperature, pH, and turbidity during purging to ensure that the groundwater level has stabilized. Generally, these measurements are made after the removal of three, four, and five well volumes.

8. If sampling using a pump, lower the pump intake to midscreen or the middle of the open section in uncased wells and collect the sample. If sampling with a bailer, lower the bailer to the sampling level before filling (this requires use of other than a "bucket-type" bailer). Purged water should be collected in a designated container and disposed of in an acceptable manner.

9. (For pump and packer assembly only). Lower assembly into well so that packer is positioned just above the screen or open section and inflate. Purge a volume equal to at least twice the screened interval or unscreened open section volume below the packer before sampling. Packers should always be tested in a casing section above ground to determine proper inflation pressures for good sealing.

10. In the event that groundwater recovery time is very slow (e.g., 24 hours), sample collection can be delayed until the following day. However, it is preferred that such a well be bailed early in the morning so that sufficient volume of water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record in the logbook.

11. Add preservative if required (see SOP F301). Label, tag, and number the sample bottle(s).

12. Volatile organics septum vials (40 ml) should be completely filled to prevent volatilization and extreme caution should be exercised when filling a vial to avoid turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a "convex meniscus." The cap is then applied and some overflow is lost, but air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles; if any are present, repeat the procedure. If the second attempt still produces air bubbles, note on Chain-of-Custody form and in field notebook and submit sample to the laboratory.

    Fill the remaining sample containers in order of decreasing volatilability (semi-volatiles next, then pesticides, PCBs, inorganics, etc.).

13. Replace the well cap. Make sure the well is readily identifiable as the source of the samples.

14. Pack the samples for shipping (see SOP F301). Attach custody seals to the shipping container. Make sure that Chain-of-Custody forms and Sample Analysis Request forms are properly filled out and enclosed or attached (see SOP F302).

15. Decontaminate all equipment.

### 5.4.2 Sample Containers

For most samples and analytical parameters, either glass or plastic containers are satisfactory. SOP F301 describes the required sampling containers for various analytes at various concentrations. Container requirements shall follow those given in USEPA Standard Operating Procedures and Quality Assurance Manual (USEPA, 1991) and SOP F301.

### 5.4.3 Preservation of Samples and Sample Volume Requirements

Sample preservation techniques and volume requirements depend on the type and concentration of the contaminant and on the type of analysis to be performed. SOP F301 describes the sample preservation and volume requirements for most of the chemicals that will be encountered during hazardous waste site investigations. Sample volume and preservation requirements shall follow those given in USEPA, 1991, and SOP F301.

### 5.4.4 Field Filtration

In general, preparation and preservation of water samples for dissolved inorganics involve some form of filtration. All samples will be filtered in the field the same day as collection. The recommended method is through the use of a disposable in-line filtration module (0.45 micron filter) utilizing the pressure provided by the upstream pumping device for its operation.

Filtration and preservation are to occur in the field on the same day as collected with the sample aliquot passing through a dedicated disposable 0.45 micron filter. Samples for organic analyses shall never be filtered.

### 5.4.5 Handling and Transporting Samples

After collection, samples should be handled as little as possible. It is preferable to use self-contained "chemical" ice (e.g., "blue ice") to reduce the risk of contamination. If water ice is used, it should be double-bagged and steps taken to ensure that the melted ice does not cause sample containers to be submerged and, thus, possibly become cross-contaminated. All sample containers should be enclosed in plastic bags or cans to prevent cross-contamination. Samples should be secured in the ice chest to prevent movement of sample containers and possible breakage. Sample packing and transportation requirements are described in SOP F301.

### 5.4.6 Sample Holding Times

Holding times (i.e., allowed time between sample collection and analysis) for routine samples are given in USEPA, 1991, and SOP F301.

## 6.0 QUALITY ASSURANCE RECORDS

Quality assurance records will be maintained for each sample that is collected. The following information will be recorded in the Field Logbook:

- Sample identification (site name, location, project no.; sample name/number and location; sample type and matrix; time and date; sampler's identity).

- Sample source and source description.
- Field observations and measurements (appearance; volatile screening; field chemistry; sampling method; volume of water purged prior to sampling; number of well volumes purged).
- Sample disposition (preservatives added; lab sent to; date and time).
- Additional remarks, as appropriate.

Proper chain-of-custody procedures play a crucial role in data gathering. SOP F302 describes the requirements for correctly completing a chain-of-custody form. Chain-of-custody forms (and sample analysis request forms) are considered quality assurance records.

## 7.0 REFERENCES

American Society of Testing and Materials. 1987. Standard Guide for Sampling Groundwater Monitoring Wells. Method D4448-85A, Annual Book of Standards, ASTM, Philadelphia, Pennsylvania.

U. S. EPA, 1991. Standard Operating Procedures and Quality Assurance Manual. Environmental Compliance Branch, U. S. EPA, Environmental Services Division, Athens, Georgia.

ATTACHMENT A

ASTM D4448-85A
STANDARD GUIDE FOR SAMPLING GROUNDWATER MONITORING WELLS

Designation: D 4448 – 85a



# Standard Guide for Sampling Groundwater Monitoring Wells[1]

This standard is issued under the fixed designation D 4448; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This guide covers procedures for obtaining valid, representative samples from groundwater monitoring wells. The scope is limited to sampling and "in the field" preservation and does not include well location, depth, well development, design and construction, screening, or analytical procedures.

1.2 This guide is only intended to provide a review of many of the most commonly used methods for sampling groundwater quality monitoring wells and is not intended to serve as a groundwater monitoring plan for any specific application. Because of the large and ever increasing number of options available, no single guide can be viewed as comprehensive. The practitioner must make every effort to ensure that the methods used, whether or not they are addressed in this guide, are adequate to satisfy the monitoring objectives at each site.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Summary of Guide

2.1 The equipment and procedures used for sampling a monitoring well depend on many factors. These include, but are not limited to, the design and construction of the well, rate of groundwater flow, and the chemical species of interest. Sampling procedures will be different if analyzing for trace organics, volatiles, oxidizable species, or trace metals is needed. This guide considers all of these factors by discussing equipment and procedure options at each stage of the sampling sequence. For ease of organization, the sampling process can be divided into three steps: well flushing, sample withdrawal, and field preparation of samples.

2.2 Monitoring wells must be flushed prior to sampling so that the groundwater is sampled, not the stagnant water in the well casing. If the well casing can be emptied, this may be done although it may be necessary to avoid oxygen contact with the groundwater. If the well cannot be emptied, procedures must be established to demonstrate that the sample represents groundwater. Monitoring an indicative parameter such as pH during flushing is desirable if such a parameter can be identified.

2.3 The types of species that are to be monitored as well as the concentration levels are prime factors for selecting sampling devices (1, 2).[2] The sampling device and all materials and devices the water contacts must be constructed of materials that will not introduce contaminants or alter the analyte chemically in any way.

2.4 The method of sample withdrawal can vary with the parameters of interest. The ideal sampling scheme would employ a completely inert material, would not subject the sample to negative pressure and only moderate positive pressure, would not expose the sample to the atmosphere, or preferably, any other gaseous atmosphere before conveying it to the sample container or flow cell for on-site analysis.

2.5 The degree and type of effort and care that goes into a sampling program is always dependent on the chemical species of interest and the concentration levels of interest. As the concentration level of the chemical species of analytical interest decreases, the work and precautions necessary for sampling are increased. Therefore, the sampling objective must clearly be defined ahead of time. For example, to prepare equipment for sampling for mg/L (ppm) levels of Total Organic Carbon (TOC) in water is about an order of magnitude easier than preparing to sample for µg/L (ppb) levels of a trace organic like benzene. The specific precautions to be taken in preparing to sample for trace organics are different from those to be taken in sampling for trace metals. No final Environmental Protection Agency (EPA) protocol is available for sampling of trace organics. A short guidance manual, (3) and an EPA document (4) concerning monitoring well sampling, including considerations for trace organics are available.

2.6 Care must be taken not to cross contaminate samples or monitoring wells with sampling or pumping devices or materials. All samples, sampling devices, and containers must be protected from the environment when not in use. Water level measurements should be made before the well is flushed. Oxidation-reduction potential, pH, dissolved oxygen, and temperature measurements and filtration should all be performed on the sample in the field, if possible. All but temperature measurement must be done prior to any significant atmospheric exposure, if possible.

2.7 The sampling procedures must be well planned and all sample containers must be prepared and labeled prior to going to the field.

## 3. Significance and Use

3.1 The quality of groundwater has become an issue of national concern. Groundwater monitoring wells are one of

[1] This guide is under the jurisdiction of ASTM Committee D-34 on Waste Disposal and is the direct responsibility of Subcommittee D34.01 on Sampling and Monitoring.

Current edition approved Aug. 23 and Oct. 25, 1985. Published May 1986.

[2] The boldface numbers in parentheses refer to a list of references at the end of this guide.

TABLE 1 Typical Container and Preservation Requirements for a Ground-Water Monitoring Program

| Sample and Measurement | Volume Required (mL) | Container P—Polyethylene G—Glass | Preservative | Maximum Holding Time |
|---|---|---|---|---|
| Metals As/Ba/Cd/Cr/Fe Pb/Sc/Ag/Mn/Na | 1000–2000 | P/G (special acid cleaning) | high purity nitric acid to pH <2 | 6 months |
| Mercury | 200–300 | P/G (special acid cleaning) | high purity nitric acid to pH <2 +0.05 % $K_2Cr_2O_7$ | 28 days |
| Radioactivity alpha/beta/radium | 4000 | P/G (special acid cleaning) | high purity nitric acid to pH <2 | 6 months |
| Phenolics | 500–1000 | G | cool, 4°C $H_2SO_4$ to pH <2 | 28 days |
| Miscellaneous | 1000–2000 | P | cool, 4°C | 28 days |
| Fluoride | 300–500 | P | | 28 days |
| Chloride | 50–200 | P/G | | 28 days |
| Sulfate | 100–500 | P/G | | 48 hours |
| Nitrate | 100–250 | P/G | | 6 h |
| Coliform | 100 | P/G | | on site/24 h |
| Conductivity | 100 | P/G | | on site/6 h |
| pH | 100 | P/G | | 48 h |
| Turbidity | 100 | P/G | | |
| Total organic carbon (TOC) | 25–100 | P/G | cool, 4°C or cool, 4°C HCl or $H_2SO_4$ to pH <2 | 24 h<br>28 days |
| Pesticides, herbicides and total organic halogen (TOX) | 1000–4000 | G/TFE-fluorocarbon lined cap solvent rinsed | cool, 4°C | 7 days/extraction +30 days/analysis |
| Extractable organics | 1000–2000 | G/TFE-fluorocarbon-lined cap solvent rinsed | cool, 4°C | 7 days/extraction +30 days/analysis |
| Organic purgeables | 25–120 | G/vial TFE-fluorocarbon-lined septum | cool, 4°C | 14 days |
| acrolein/acrylonitrile | | | | 3 days |

e more important tools for evaluating the quality of oundwater, delineating contamination plumes, and estabhing the integrity of hazardous material management cilities.

3.2 The goal in sampling groundwater monitoring wells is obtain samples that are truly representative of the aquifer groundwater in question. This guide discusses the advanges and disadvantages of various well flushing, sample thdrawal, and sample preservation techniques. It reviews parameters that need to be considered in developing a id sampling plan.

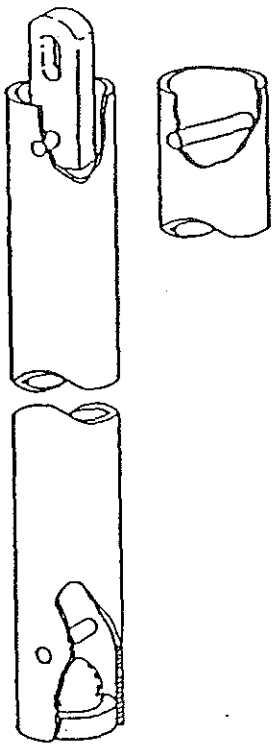
## Well Flushing (Purging)

4.1 Water that stands within a monitoring well for a long iod of time may become unrepresentative of formation ter because chemical or biochemical change may cause er quality alterations and even if it is unchanged from the e it entered the well, the stored water may not be resentative of formation water at the time of sampling, or h. Because the representativeness of stored water is stionable, it should be excluded from samples collected n a monitoring well.

4.2 The surest way of accomplishing this objective is to ove all stored water from the casing prior to sampling. earch with a tracer in a full scale model 2 in. PVC well (5) cates that pumping 5 to 10 times the volume of the well an inlet near the free water surface is sufficient to remove he stored water in the casing. The volume of the well may be calculated to include the well screen and any gravel pack if natural flow through these is deemed insufficient to keep them flushed out.

4.3 In deep or large diameter wells having a volume of water so large as to make removal of all the water impractical, it may be feasible to lower a pump or pump inlet to some point well below the water surface, purge only the volume below that point then withdraw the sample from a deeper level. Research indicates this approach should avoid most contamination associated with stored water (5, 6, 7). Sealing the casing above the purge point with a packer may make this approach more dependable by preventing migration of stored water from above. But the packer must be above the top of the screened zone, or stagnant water from above the packer will flow into the purged zone through the well's gravel/sand pack.

4.4 In low yielding wells, the only practical way to remove all standing water may be to empty the casing. Since it is not always possible to remove all water, it may be advisable to let the well recover (refill) and empty it again at least once. If introduction of oxygen into the aquifer may be of concern, it would be best not to uncover the screen when performing the above procedures. The main disadvantage of methods designed to remove all the stored water is that large volumes may need to be pumped in certain instances. The main advantage is that the potential for contamination of samples with stored water is minimized.



NOTE—Taken from Ref (15).

FIG. 1 Single Check Valve Bailer

nother approach to well flushing is to monitor one indicator parameters such as pH, temperature, or nductivity and consider the well to be flushed when the dicator(s) no longer change. The advantage of this method that pumping can be done from any location within the sing and the volume of stored water present has no direct aring on the volume of water that must be pumped. bviously, in a low yielding well, the well may be emptied fore the parameters stabilize. A disadvantage of this proach is that there is no assurance in all situations that e stabilized parameters represent formation water. If signifint drawdown has occurred, water from some distance ay may be pulled into the screen causing a steady rameter reading but not a representative reading. Also, a itable indicator parameter and means of continuously easuring it in the field must be available.

4.6 Gibb (4, 8) has described a time-drawdown approach ing a knowledge of the well hydraulics to predict the rcentage of stored water entering a pump inlet near the top the screen at any time after flushing begins. Samples are ken when the percentage is acceptably low. As before, the vantage is that well volume has no direct effect in the ration of pumping. A current knowledge of the well's draulic characteristics is necessary to employ this apoach. Downward migration of stored water due to effects ier than drawdown (for example density differences) is not nted for in this approach.

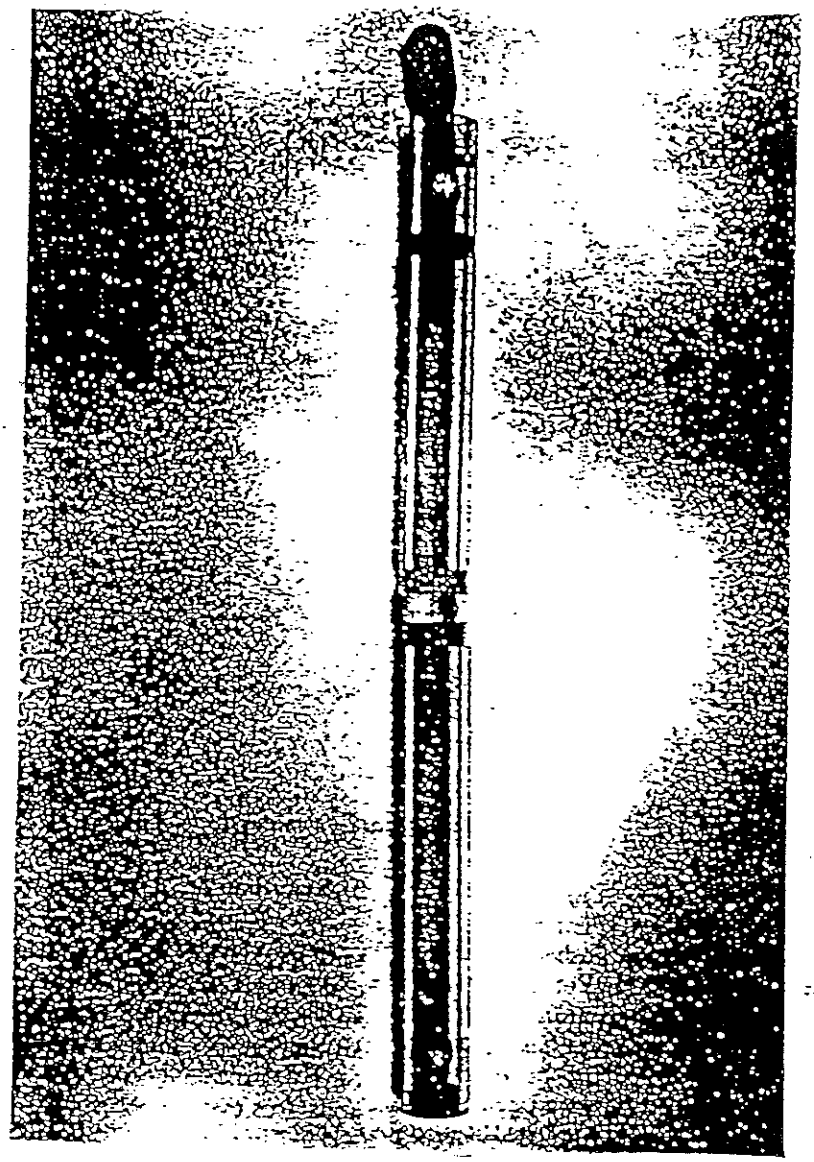
any flushing approach, a withdrawal rate that drawdown while satisfying time constraints ould be used. Excessive drawdown distorts the natural flow terns around a well and can cause contaminants that were t present originally to be drawn into the well.

## 5. Materials and Manufacture

5.1 The choice of materials used in the construction of sampling devices should be based upon a knowledge of what compounds may be present in the sampling environment and how the sample materials may interact via leaching, adsorption, or catalysis. In some situations, PVC or some other plastic may be sufficient. In others, an all glass apparatus may be necessary.

5.2 Most analytical protocols suggest that the devices used in sampling and storing samples for trace organics analysis (μg/L levels) must be constructed of glass or TFE-fluorocarbon resin, or both. One suggestion advanced by the EPA is that the monitoring well be constructed so that only TFE–fluorocarbon tubing be used in that portion of the sampling well that extends from a few feet above the water table to the bottom of the borehole. (3, 5) Although this type of well casing is now commercially available, PVC well casings are currently the most popular. If adhesives are avoided, PVC well casings are acceptable in many cases although their use may still lead to some problems if trace organics are of concern. At present, the type of background presented by PVC and interactions occurring between PVC and groundwater are not well understood. Tin, in the form of an organotin stabilizer added to PVC, may enter samples taken from PVC casing. (9)

5.3 Since the most significant problem encountered in trace organics sampling, results from the use of PVC adhesives in monitoring well construction, threaded joints might avoid the problem (3, 5). Milligram per litre (parts per million) levels of compounds such as tetrahydrofuran, methyl-ethyl-ketone, and toluene are found to leach into



NOTE—Taken from Ref (17).

FIG. 2 Acrylic Point Source Bailer

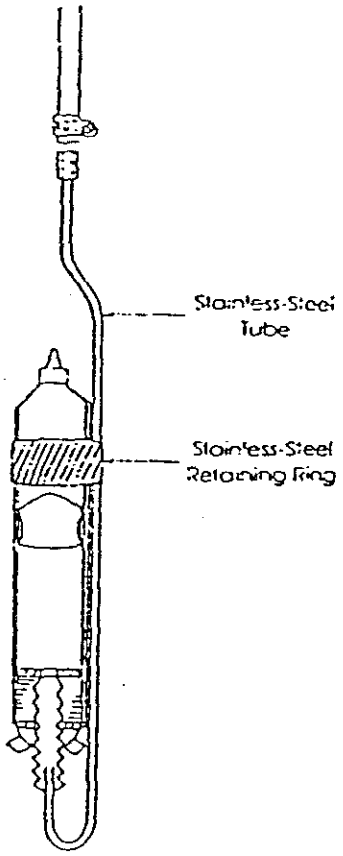
groundwater samples from monitoring well casings sealed with PVC solvent cement. Pollutant phthalate esters (8, 10) are often found in water samples at ppb levels; the EPA has found them on occasion at ppm levels in their samples. The ubiquitous presence of these phthalate esters is unexplained, except to say that they may be leached from plastic pipes, sampling devices, and containers.

5.4 TFE-fluorocarbon resins are highly inert and have sufficient mechanical strength to permit fabrication of sampling devices and well casings. Molded parts are exposed to high temperature during fabrication which destroys any organic contaminants. The evolution of fluorinated compounds can occur during fabrication, will cease rapidly, and does not occur afterwards unless the resin is heated to its melting point.

5.5 Extruded tubing of TFE-fluorocarbon for sampling may contain surface traces of an organic solvent extrusion aid. This can be removed easily by the fabricator and, once removed by flushing, should not affect the sample. TFE-fluorocarbon FEP and TFE-fluorocarbon PFA resins do not require this extrusion aid and may be suitable for sample tubing as well. Unsintered thread-sealant tape of TFE-fluorocarbon is available in an "oxygen service" grade and contains no extrusion aid and lubricant.

5.6 Louneman, et al. (11) alludes to problems caused by a lubricating oil used during TFE-fluorocarbon tubing extrusion. This reference also presents evidence that a fluorinated ethylene-propylene copolymer adsorbed acetone to a degree that later caused contamination of a gas sample.

5.7 Glass and stainless steel are two other materials generally considered inert in aqueous environments. Glass is probably among the best choices though it is not inconceivable it could adsorb some constituents as well as release other contaminants (for example, Na, silicate, and Fe). Of course, glass sampling equipment must be handled carefully in the field. Stainless steel is strongly and easily machined to




DTE—Taken from Ref (21).

FIG. 3 Schematic of the Inverted Syringe Sampler

fabricate equipment. Unfortunately, it is not totally immune to corrosion that could release metallic contaminants. Stainless steel contains various alloying metals, some of these (for example Ni) are commonly used as catalysts for various reactions. The alloyed constituents of some stainless steels can be solubilized by the pitting action of nonoxidizing anions such as chloride, fluoride, and in some instances sulfate, over a range of pH conditions. Aluminum, titanium, polyethylene, and other corrosion resistant materials have been proposed by some as acceptable materials, depending on groundwater quality and the constituents of interest.

5.8 Where temporarily installed sampling equipment is used, the sampling device that is chosen should be nonplastic (unless TFE-fluorocarbon), cleanable of trace organics, and must be cleaned between each monitoring well use in order to avoid cross-contamination of wells and samples. The only way to ensure that the device is indeed "clean" and acceptable is to analyze laboratory water blanks and field water blanks that have been soaked in and passed through the sampling device to check for the background levels that may result from the sampling materials or from field conditions. Thus, all samplings for trace materials should be accompanied by samples which represent the field background (if possible), the sampling equipment background, and the laboratory background.

5.9 Additional samples are often taken in the field and spiked (spiked-field samples) in order to verify that the sample handling procedures are valid. The American Chemical Society's committee on environmental improvement h published guidelines for data acquisition and data evaluati which should be useful in such environmental evaluatio (10, 12).

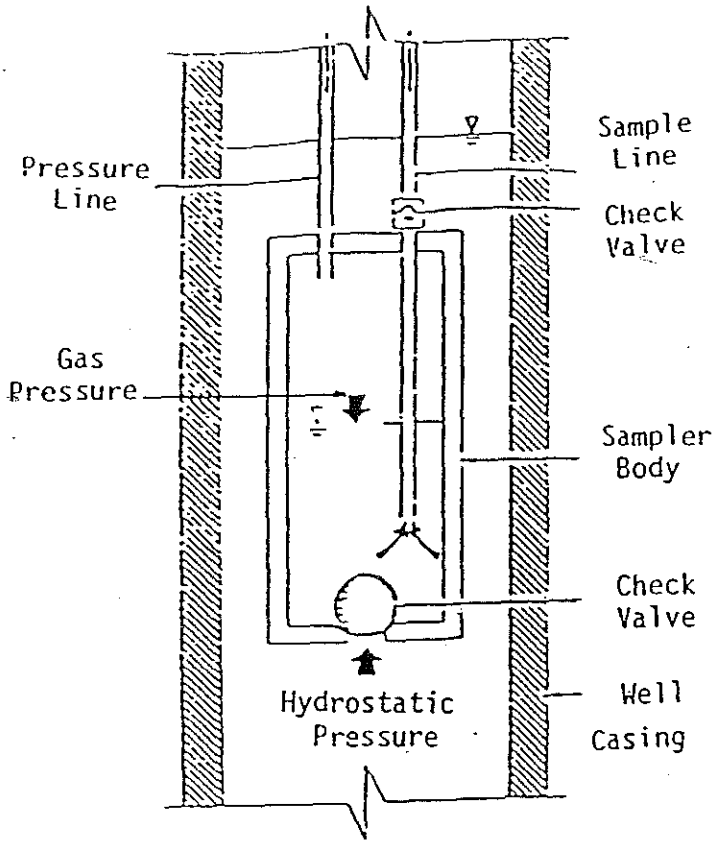
## 6. Sampling Equipment

6.1 There is a fairly large choice of equipment present available for groundwater sampling from single screen wells and well clusters. The sampling devices can be categ rized into the following eight basic types.

6.1.1 *Down-Hole Collection Devices:*

6.1.1.1 Bailers, messenger bailers, or thief samplers (1 14) are examples of down-hole devices that probably provi valid samples once the well has been flushed. They are n practical for removal of large volumes of water. The devices can be constructed in various shapes and sizes from variety of materials. They do not subject the sample t pressure extremes.

6.1.1.2 Bailers do expose part of the sample to th atmosphere during withdrawal. Bailers used for sampling volatile organic compounds should have a sample cock draft valve in or near the bottom of the sampler allowir withdrawal of a sample from the well below the expose surface of the water or the first few inches of the samp should be discarded. Suspension lines for bailers and oth samplers should be kept off the ground and free of oth contaminating materials that could be carried into the wel Down-hole devices are not very practical for use in dee




NOTE—Taken from Ref (5).

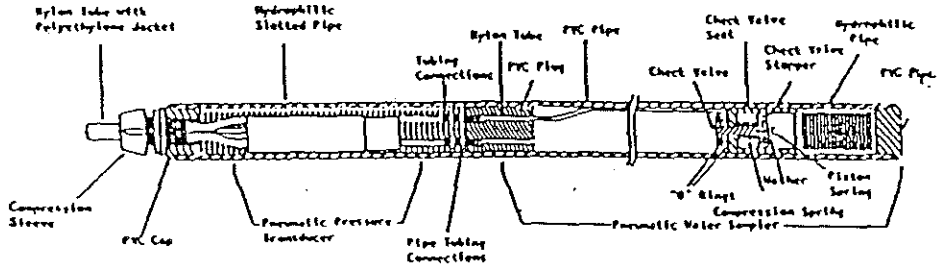
FIG. 4 The Principal of Gas Displacement Pumping

cells. However, potential sample oxidation during transfer of ie sample into a collection vessel and time constraints for wering and retrieval for deep sampling are the primary sadvantages.

6.1.1.3 Three down-hole devices are the single and double eck valve bailers and thief samplers. A schematic of a ngle check valve unit is illustrated in Fig. 1. The bailer may threaded in the middle so that additional lengths of blank sing may be added to increase the sampling volume. E-fluorocarbon or PVC are the most common materials ed for construction (15).

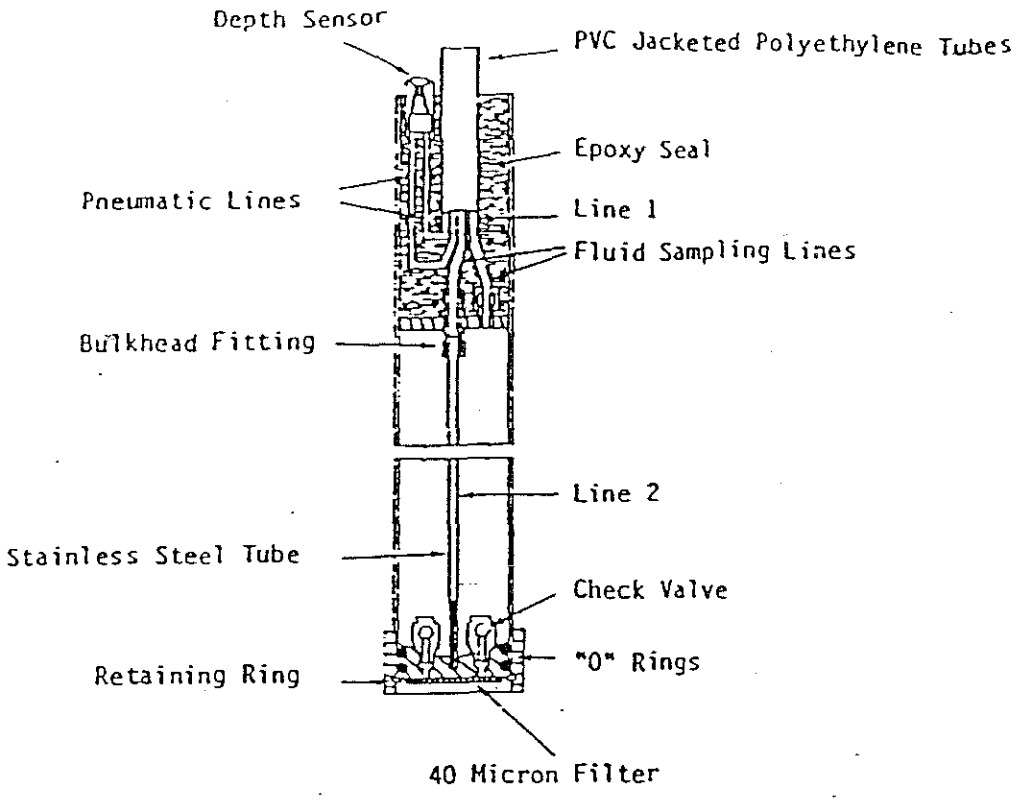
6.1.1.4 In operation, the single check valve bailer is wered into the well, water enters the chamber through the ttom, and the weight of the water column closes the check valve upon bailer retrieval. The specific gravity of the ball should be about 1.4 to 2.0 so that the ball almost sits on the check valve seat during chamber filling. Upon bailer withdrawal, the ball will immediately seat without any samples loss through the check valve. A similar technique involves lowering a sealed sample container within a weighted bottle into the well. The stopper is then pulled from the bottle via a line and the entire assembly is retrieved upon filling of the container (14, 16).

6.1.1.5 A double check valve bailer allows point source sampling at a specific depth (15, 17). An example is shown in Fig. 2. In this double check valve design, water flows through the sample chamber as the unit is lowered. A venturi tapered inlet and outlet ensures that water passes freely through the




NOTE—Taken from Ref (41).

FIG. 5 Pneumatic Water Sampler With Internal Transducer




NOTE—Taken from Ref (42).

FIG. 6 Pneumatic Sampler With Externally Mounted Transducer

unit. When a depth where the sample is to be collected is reached, the unit is retrieved. Because the difference between each ball and check valve seat is maintained by a pin that blocks vertical movement of the check ball, both check valves close simultaneously upon retrieval. A drainage pin is placed into the bottom of the bailer to drain the sample directly into a collection vessel to reduce the possibility of air oxidation. The acrylic model in Fig. 2 is threaded at the midsection allowing the addition of threaded casing to increase the sampling volume.

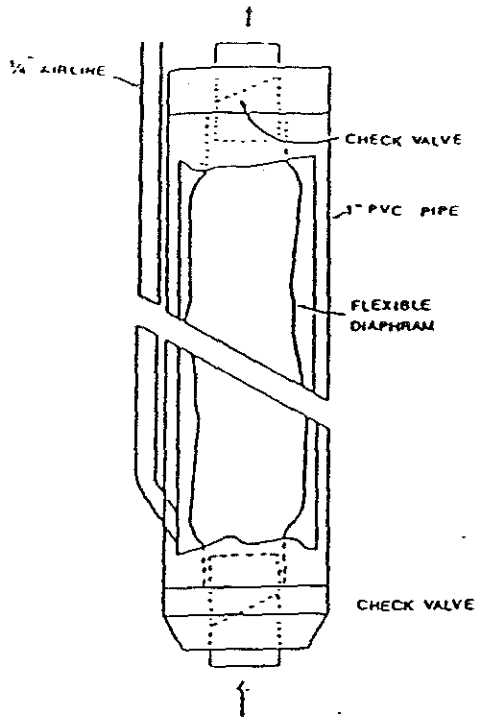
6.1.1.6 Another approach for obtaining point source samples employs a weighted messenger or pneumatic change to "trip" plugs at either end of an open tube (for example, tube water sampler or thief sampler) to close the chamber (18). Foerst, Kemmerer, and Bacon samplers are of this variety (14, 17, 19). A simple and inexpensive pneumatic sampler was recently described by Gillham (20). The device (Fig. 3) consists of a disposable 50 mL plastic syringe modified by sawing off the plunger and the finger grips. The syringe is then attached to a gas-line by means of a rubber stopper assembly. The gas-line extends to the surface, and is used to drive the stem-less plunger, and to raise and lower the syringe into the hole. When the gas-line is pressurized, the rubber plunger is held at the tip of the syringe. The sampler is then lowered into the installation, and when the desired depth is reached, the pressure in the gas-line is reduced to atmospheric (or slightly less) and water enters the syringe. The sampler is then retrieved from the installation and the syringe detached from the gas-line. After the tip is sealed, the syringe is used as a short-term storage container. A number of thief or messenger devices are available in various materials and shapes.

6.1.2 *Suction Lift Pumps:*

6.1.2.1 Three types of suction lift pumps are the direct line, centrifugal, and peristaltic. A major disadvantage of any suction pump is that it is limited in its ability to raise water by the head available from atmospheric pressure. Thus, if the surface of the water is more than about 25 ft below the pump, water may not be withdrawn. The theoretical suction limit is about 34 ft, but most suction pumps are capable of maintaining a water-lift of only 25 ft or less.

6.1.2.2 Many suction pumps draw the water through some sort of volute in which impellers, pistons, or other devices operate to induce a vacuum. Such pumps are probably unacceptable for most sampling purposes because they are usually constructed of common materials such as brass or mild steel and may expose samples to lubricants. They often induce very low pressures around rotating vanes or other such parts such that degassing or even cavitation may occur. They can mix air with the sample via small leaks in the casing, and they are difficult to adequately clean between uses. Such pumps are acceptable for purging of wells, but should not generally be used for sampling.

6.1.2.3 One exception to the above statements is a peristaltic pump. A peristaltic pump is a self-priming, low volume suction pump which consists of a rotor with ball bearing rollers (21). Flexible tubing is inserted around the pump rotor and squeezed by heads as they revolve in a circular pattern around the rotor. One end of the tubing is placed into the well while the other end can be connected




NOTE—Taken from Ref (4).

FIG. 7 Bladder Pump

rectly to a receiving vessel. As the rotor moves, a reduced essure is created in the well tubing and an increased essure (<40 psi) on the tube leaving the rotor head. A drive aft connected to the rotor head can be extended so that ultiple rotor heads can be attached to a single drive shaft.

6.1.2.4 The peristaltic pump moves the liquid totally thin the sample tube. No part of the pump contacts the uid. The sample may still be degassed (cavitation is likely) but the problems due to contact with the pump chanism are eliminated. Peristaltic pumps do require a rly flexible section of tubing within the pumphead itself. A tion of silicone tubing is commonly used within the istaltic pumphead, but other types of tubing can be used ticularly for the sections extending into the well or from pump to the receiving container. The National Council he Paper Industry for Air and Stream Improvement (22) ommends using medical grade silicone tubing for organic mpling purposes as the standard grade uses an organic canizing agent which has been shown to leach into mples. Medical grade silicone tube is, however, limited to over a restricted range of ambient temperatures. Various nufacturers offer tubing lined with TFE-fluorocarbon or on[3] for use with their pumps. Gibb (1, 8) found little erence between samples withdrawn by a peristaltic pump those taken by a bailer.

1.2.5 A direct method of collecting a sample by suction sists of lowering one end of a length of plastic tubing into well or piezometer. The opposite end of the tubing is nected to a two way stopper bottle and a hand held or mechanical vacuum pump is attached to a second tubing leaving the bottle. A check valve is attached between the two lines to maintain a constant vacuum control. A sample can then be drawn directly into the collection vessel without contacting the pump mechanism (5, 23, 24).

6.1.2.6 A centrifugal pump can be attached to a length of plastic tubing that is lowered into the well. A foot valve is usually attached to the end of the well tubing to assist in priming the tube. The maximum lift is about 4.6 m (15 ft) for such an arrangement (23, 25, 26).

6.1.2.7 Suction pump approaches offer a simple sample retrieval method for shallow monitoring. The direct line method is extremely portable though considerable oxidation and mixing may occur during collection. A centrifugal pump will agitate the sample to an even greater degree although pumping rates of 19 to 151 Lpm (5 to 40 gpm) can be attained. A peristaltic pump provides a lower sampling rate with less agitation than the other two pumps. The withdrawal rate of peristaltic pumps can be carefully regulated by adjustment of the rotor head revolution.

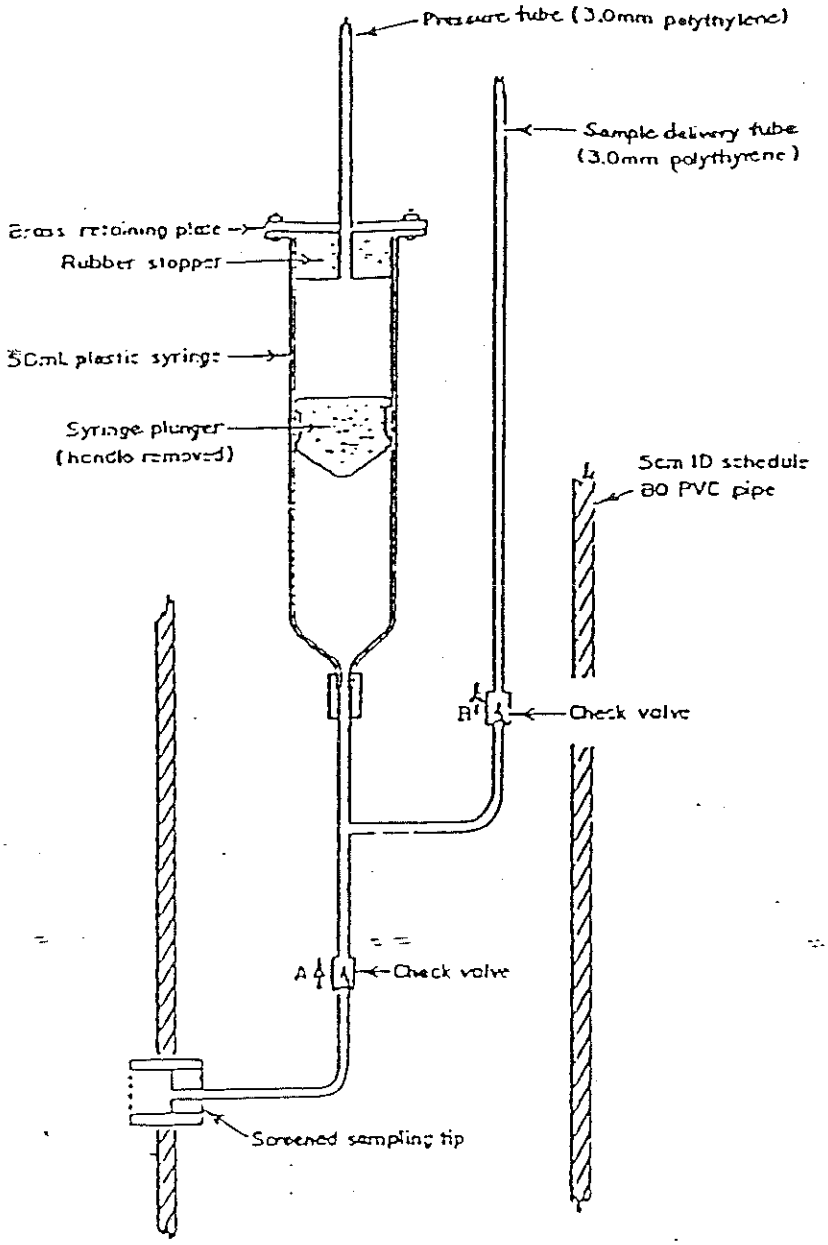
6.1.2.8 All three systems can be specially designed so that the water sample contacts only the TFE flourocarbon or silicone tubing prior to sample bottle entry. Separate tubing is recommended for each well or piezometer sampled.

6.1.3 *Electric Submersible Pumps:*

6.1.3.1 A submersible pump consists of a sealed electric motor that powers a piston or helical single thread worm at a high rpm. Water is brought to the surface through an access tube. Such pumps have been used in the water well industry for years and many designs exist (5, 26).

6.1.3.2 Submersible pumps provide relatively high discharge rates for water withdrawal at depths beyond suction

[3] iton is a trademark of E. I. du Pont de Nemours & Co., Wilmington, DE and has been found suitable for this purpose.




NOTE—Taken from Ref (48).

FIG. 8 Positive Displacement Syringe Pump

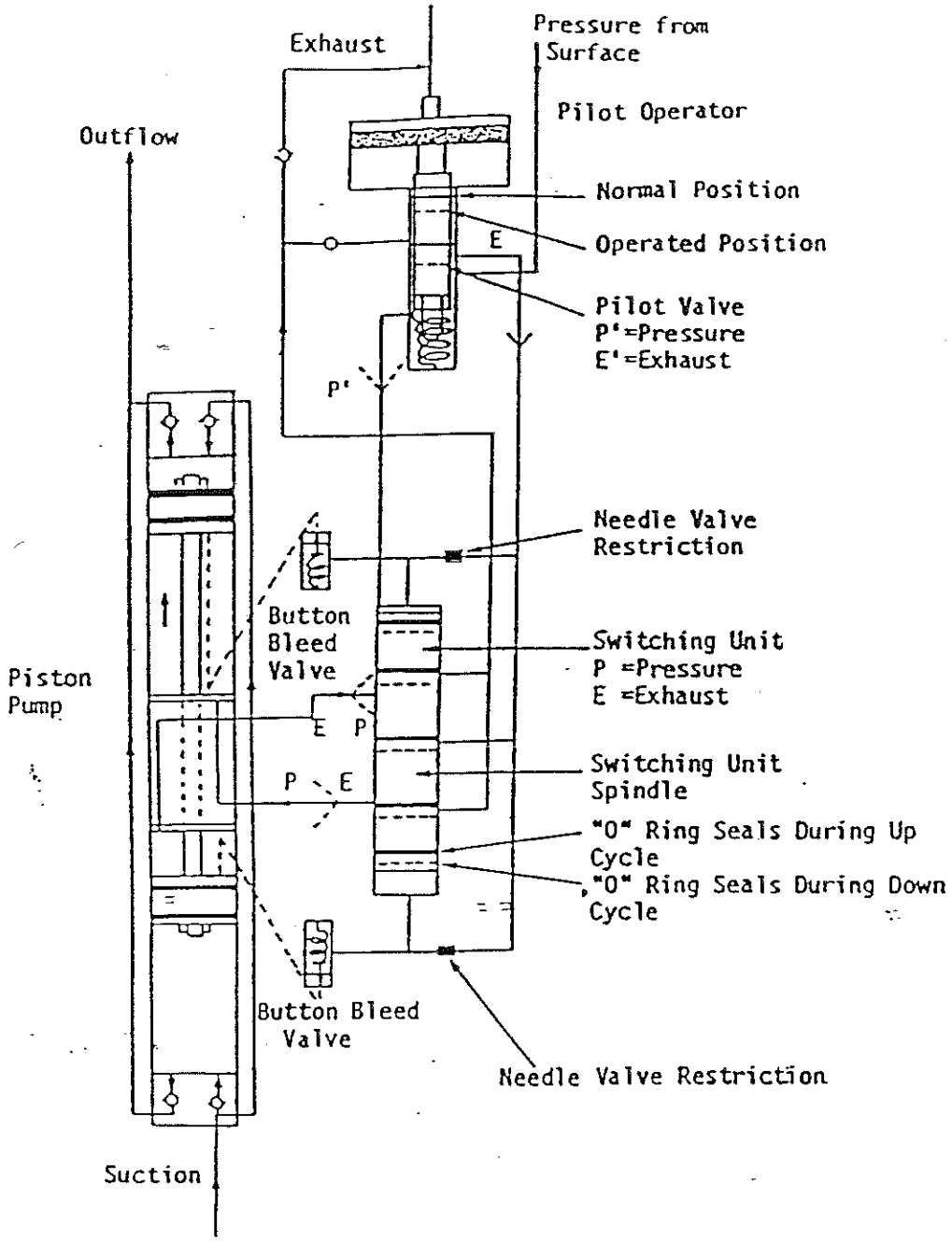
lift capabilities. A battery operated unit 3.6 cm (1.4 in.) in diameter and with a 4.5 Lpm (1.2 gpm) flow rate at 33.5 m (110 ft) has been developed (27). Another submersible pump has an outer diameter of 11.4 cm (4.5 in.) and can pump water from 91 m (300 ft). Pumping rates vary up to 53.0 Lpm (14 gpm) depending upon the depth of the pump (28).

6.1.3.3 A submersible pump provides higher extraction rates than many other methods. Considerable sample agitation results, however, in the well and in the collection tube during transport. The possibility of introducing trace metals to the sample from pump materials also exists. Steam cleaning of the unit followed by rinsing with unchlorinated, deionized water is suggested between sampling when analysis for organics in the parts per million (ppm) or parts per billion (ppb) range is required (29).

6.1.4 *Gas-Lift Pumps:*

6.1.4.1 Gas-lift pumps use compressed air to bring a water sample to the surface. Water is forced up an eductor pipe that may be the outer casing or a smaller diameter pipe inserted into the well annulus below the water level (30, 31).

6.1.4.2 A similar principle is used for a unit that consists of a small diameter plastic tube perforated in the lower end. This tube is placed within another tube of slightly larger diameter. Compressed air is injected into the inner tube; the air bubbles through the perforations, thereby lifting the water sample via the annulus between the outer and inner tubing (32). In practice, the eductor line should be submerged to a depth equal to 60 % of the total submerged eductor length during pumping (26). A 60 % ratio is considered optimal although a 30 % submergence ratio is adequate.




NOTE—Taken from Ref (49).

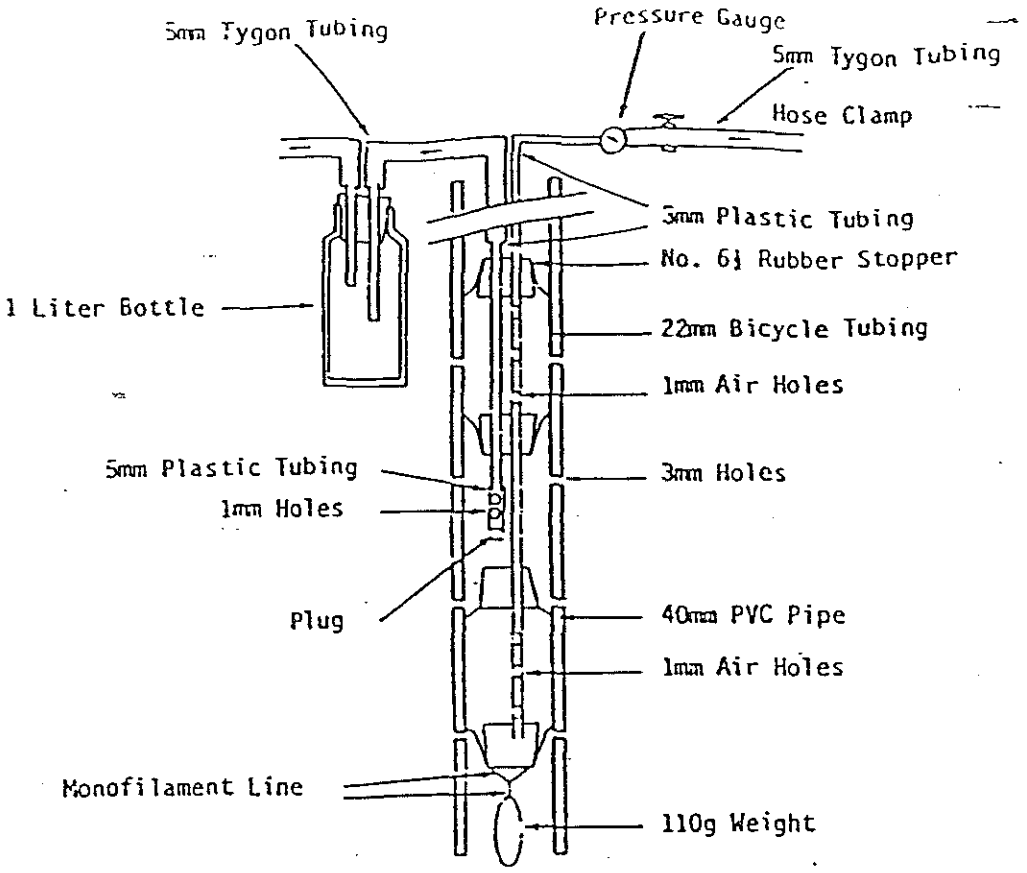
FIG. 9 Gas Driven Piston Pump

6.1.4.3 The source of compressed gas may be a hand ımp for depths generally less than 7.6 m (25 ft). For greater :pths, air compressors, pressurized air bottles, and air ımpressed from an automobile engine have been used.

6.1.4.4 As already mentioned, gas-lift methods result in ınsiderable sample agitation and mixing within the well, ıd cannot be used for samples which will be tested for ılatile organics. The eductor pipe or weighted plastic tubing a potential source of sample contamination. In addition, ibb (8) uncovered difficulties in sampling for inorganics. hese difficulties were attributed to changes in redox, pH, and species transformation due to solubility constant changes resulting from stripping, oxidation, and pressure changes.

6.1.5 *Gas Displacement Pumps:*

6.1.5.1 Gas displacement or gas drive pumps are distinguished from gas-lift pumps by the method of sample transport. Gas displacement pumps force a discrete column of water to the surface via mechanical lift without extensive mixing of the pressurized gas and water as occurs with air-lift equipment. The principle is shown schematically in Fig. 4. Water fills the chamber. A positive pressure is applied to the




E—Taken from Ref (53).

FIG. 10 Packer Pump Arrangement

gas line closing the sampler check valve and forcing water up the sample line. By removing the pressure the cycle can be repeated. Vacuum can also be used in conjunction with the gas (30). The device can be permanently installed in the well (33, 34, 35) or lowered into the well (36, 37).

6.1.5.2 A more complicated two stage design constructed of glass with check valves made of TFE-fluorocarbon has been constructed (38, 39). The unit was designed specifically for sample testing for trace level organics. Continuous flow rates up to 2.3 Lpm (0.6 gpm) are possible with a 5.1 cm (2 in.) diameter unit.

6.1.5.3 Gas displacement pumps have also been developed with multiple functions. The water sample in Fig. 5 provides piezometric data measurements with an internally mounted transducer (40). A sample with its transducer exposed externally for piezometric measurements is illustrated in Fig. 6 (41). The sensor can activate the gas source at the surface to cause sample chamber pressurization at the predetermined depth. Another design can be used as a water sampler or as a tool for injecting brine or other tracers into a well (42).

6.1.5.4 Gas displacement pumps offer reasonable potential for preserving sample integrity because little of the driving gas comes in contact with the sample as the sample is conveyed to the surface by a positive pressure. There is, however, a potential loss of dissolved gasses or contamination from the driving gas and the housing materials.

6.1.6 *Bladder Pumps:*

6.1.6.1 Bladder pumps, also referred to as gas-operated squeeze pumps, consist of a flexible membrane enclosed by a rigid housing. Water enters the membrane through a check valve in the vessel bottom; compressed gas injected into the cavity between the housing and bladder forces the sample through a check valve at the top of the membrane and into a discharge line (Fig. 7). Water is prevented from re-entering the bladder by the top check valve. The process is repeated to cycle the water to the surface. Samples taken from depths of 30.5 m (100 ft) have been reported.

6.1.6.2 A variety of design modifications and materials are available (43, 44). Bladder materials include neoprene, rubber, ethylene propylene terpolymer (E.P.T.), nitrile, and the fluorocarbon Viton.[3] A bladder made of TFE-fluorocarbon is also under development (45). Automated sampling systems have been developed to control the time between pressurization cycles (46).

6.1.6.3 Bladder pumps provide an adaptable sampling tool due primarily to the number of bladder shapes that are feasible. These devices have a distinct advantage over gas displacement pumps in that there is no contact with the driving gas. Disadvantages include the large gas volumes required, low pumping rates, and potential contamination from many of the bladder materials, the rigid housing, or both.

6.1.7 *Gas Driven Piston Pumps:*

6.1.7.1 A simple and inexpensive example of a gas driven piston pump is a syringe pump (47). The pump (Fig. 8) is constructed from a 50 mL plastic syringe with plunger stem removed. The device is connected to a gas line to the surface and the sample passes through a check valve arrangement to a sampling container at the surface. By successively applying positive and negative pressure to the gas-line, the plunger is activated driving water to the surface.

6.1.7.2 A double piston pump powered by compressed air is illustrated in Fig. 9. Pressurized gas enters the chamber between the pistons; the alternating chamber pressurization activates the piston which allows water entry during the suction stroke of the piston and forces the sample to the surface during the pressure stroke (48). Pumping rates between 9.5 and 30.3 L/hr (2.5 to 8 gal/hr) have been reported from 30.5 m (100 ft). Depths in excess of 457 m (1500 ft) are possible.

6.1.7.3 The gas piston pump provides continuous sample withdrawal at depths greater than is possible with most other approaches. Nevertheless, contribution of trace elements from the stainless steel and brass is a potential problem and the quantity of gas used is significant.

6.1.8 *Packer Pump Arrangement:*

6.1.8.1 A packer pump arrangement provides a means by which two expandable "packers" isolate a sampling unit between two packers within a well. Since the hydraulic or pneumatic activated packers are wedged against the casing wall or screen, the sampling unit will obtain water samples only from the isolated well portion. The packers are deflated for vertical movement within the well and inflated when the desired depth is attained. Submersible, gas lift, and suction pumps can be used for sampling. The packers are usually constructed from some type of rubber or rubber compound (48, 49, 50, 51). A packer pump unit consisting of a vacuum sampler positioned between two packers is illustrated in Fig. 10 (52).

6.1.8.2 A packer assembly allows the isolation of discrete sampling points within a well. A number of different samplers can be situated between the packers depending upon the analytical specifications for sample testing. Vertical movement of water outside the well casing during sampling is possible with packer pumps but depends upon the pumping rate and subsequent disturbance. Deterioration of the expandable materials will occur with time with the increased possibility of undesirable organic contaminants contributing to the water sample.

## 7. Sample Containers and Preservation

7.1 Complete and unequivocal preservation of samples, whether domestic wastewater, industrial wastes, or natural waters, is practically impossible. At best, preservation techniques only retard the chemical and biological changes that inevitably continue after the sample is removed from the source. Therefore, insuring the timely analysis of a sample should be one of the foremost considerations in the sampling plan schedule. Methods of preservation are somewhat limited and are intended to retard biological action, retard hydrolysis of chemical compounds and complexes, and reduce the volatility of constituents. Preservation methods are generally limited to pH control, chemical addition, refrigeration and freezing. For water samples, immediate refrigeration just above freezing (4°C in wet ice) is often the best preservation technique available, but it is not the only measure nor is it applicable in all cases. There may be special cases where it might be prudent to include a recording thermometer in the sample shipment to verify the maximum and minimum temperature to which the samples were exposed. Inexpensive devices for this purpose are available.

7.2 All bottles and containers must be specially precleaned, pre-labelled, and organized in ice-chests (isolating samples and sampling equipment from the environment) before one goes into the field. Otherwise, in any comprehensive program utter chaos usually develops in the field or laboratory. The time in the field is very valuable and should be spent on taking field notes, measurements, and in documenting samples, not on labelling and organizing samples. Therefore, the sampling plan should include clear instructions to the sampling personnel concerning the information required in the field data record logbook (notebook), the information needed on container labels for identification, the chain-of-custody protocols, and the methods for preparing field blanks and spiked samples. Example of detailed plans and documentation procedures have been published (14, 53).

7.3 The exact requirements for the volumes of sample needed and the number of containers to use may vary from laboratory to laboratory. This will depend on the specific analyses to be performed, the concentration levels of interest, and the individual laboratory protocols. The manager of the sampling program should make no assumptions about the laboratory analyses. He should discuss the analytical requirements of the sampling program in detail with the laboratory coordinator beforehand. This is especially the case since some analyses and preservation measures must be performed at the laboratory as soon as possible after the samples arrive. Thus, appropriate arrangements must be made.

7.4 There are a number of excellent references available which list the containers and preservation techniques appropriate for water and soils (13, 14, 50, 54, 55, 56). The "Handbook for Sampling and Sample Preservation of Water and Wastewater" is an excellent reference and perhaps the most comprehensive one (14). Some of this information is summarized in Table 1.

7.5 Sample containers for trace organic samples require special cleaning and handling considerations (57). The sample container for purgeable organics consist of a screw-cap vial (25 to 125 mL) fitted with a TFE-flourocarbon faced silicone septum. The vial is sealed in the laboratory immediately after cleaning and is only opened in the field just prior to pouring sample into it. The water sample then must be sealed into the vial headspace free (no air bubbles) and immediately cooled (4°C) for shipment. Multiple samples (usually about four taken from one large sample container) are taken because leakage of containers may cause losses, may allow air to enter the containers, and may cause erroneous analysis of some constituents. Also, some analyses are best conducted on independent protected samples.

7.6 The purgeable samples must be analyzed by the laboratory within 14 days after collection, unless they are to be analyzed for acrolein or acrylonitrile (in which case they are to be analyzed within 3 days). For samples for solvent extractions (extractable organics-base neutrals, acids and

pesticides), the sample bottles are narrow mouth, screw cap quart bottles or half-gallon bottles that have been precleaned, rinsed with the extracting organic solvent and oven dried at C for at least 1 h. These bottles must be sealed with ...-fluorocarbon lined caps (Note). Samples for organic extraction must be extracted within 7 days and analyzed within 30 days after extraction. Special pre-cleaned, solvent rinsed and oven-dried stainless steel beakers (one for each monitoring well) may be used for transferring samples from the sampling device to the sample containers.

NOTE—When collecting samples, the bottles should not be overfilled or prerinsed with sample before filling because oil and other materials may remain in the bottle. This can cause erroneously high results.

7.7 For a number of groundwater parameters, the most meaningful measurements are those made in the field at the time of sample collection or at least at an on-site laboratory. These include the water level in the well and parameters that sometimes can change rapidly with storage. A discussion of the various techniques for measuring the water level in the well is contained in a NCASI publication (5) and detailed procedures are outlined in a U.S. Geological Survey publication (58). Although a discussion of these techniques is beyond the scope of this guide, it is important to point out that accurate measurements must be made before a well is flushed or only after it has had sufficient time to recover. Parameters that can change rapidly with storage include specific conductance, pH, turbidity, redox potential, dissolved oxygen, and temperature. For some of the other parameters, the emphasis in groundwater monitoring is on the concentration of each specific dissolved component, not the total concentration of each. Samples for these types of measurements should be filtered through 0.45 μm membrane filters ideally in the field or possibly at an on-site laboratory as soon as possible. Analyses often requiring filtered samples include all metals, radioactivity parameters, total organic carbon, dissolved orthophosphate (if needed), and total dissolved phosphorous (if needed) (13, 14). If metals are to be analyzed, filter the sample prior to acid preservation. For TOC organics, the filter material should be tested to assure that it does not contribute to the TOC. The type or size of the filter to be used is not well understood. However, if results of metal, TOC or other parameters that could be effected by solids are to be compared, the same filtering procedure must be used in each case. Repeated analytical results should state whether the samples were filtered and how they were filtered.

7.8 Shipment and receipt of samples must be coordinated with the laboratory to minimize time in transit. All samples for organic analysis (and many other parameters), should arrive at the laboratory within one day after it is shipped and be maintained at about 4°C with wet ice. The best way to get them to the laboratory in good condition is to send them in sturdy insulated ice chests (coolers) equipped with bottle dividers. 24-h courier service is recommended, if personal delivery service is not practical.

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# F105
# *SURFACE WATER AND SEDIMENT SAMPLE ACQUISITION*

# SURFACE WATER AND SEDIMENT SAMPLE ACQUISITION

## 1.0 PURPOSE

This procedure describes methods and equipment commonly used for collecting environmental samples of surface water and aquatic sediment either for on-site examination and chemical testing or for laboratory analysis.

## 2.0 SCOPE

The information presented in this SOP is generally applicable to all environmental sampling of surface waters (Section 5.2) and aquatic sediments (Section 5.3), except where the analyte(s) may interact with the sampling equipment.

Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be documented and presented in the Sampling and Analysis Plan.

## 3.0 DEFINITIONS

Grab Sample - An individual sample collected from a single location at a specific time or period of time generally not exceeding 15 minutes.

Composite Sample - A sample collected over time that typically consists of a series of discrete samples which are combined or composited.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other, approved procedures are developed. The Project Manager is responsible for development of documentation for procedures which deviate from those presented herein.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the specific surface water and/or sediment sampling techniques and equipment to be used, and documenting these in accordance with the Sampling and Analysis Plan. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and that personnel performing sampling activities have been briefed and trained to execute these procedures.

Sampling Personnel - It is the responsibility of the field sampling personnel to follow these procedures, or to follow documented, project-specific procedures as directed by the Field Team Leader and/or the Project Manager. The sampling personnel are responsible for the proper acquisition of surface water and sediment samples.

## 5.0 PROCEDURES

Collecting a representative sample from surface water or sediments is difficult due to water movement, stratification or patchiness. To collect representative samples, one must standardize sampling bias related to site selection; sampling frequency; sample collection; sampling devices; and sample handling, preservation, and identification.

Representativeness is a qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important quality not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location, selection, and collection methods are important to ensure that a truly representative sample has been collected. Regardless of scrutiny and quality control applied during laboratory analyses, reported data are only as good as the confidence that can be placed on the representativeness of the samples.

### 5.1 Defining the Sampling Program

Many factors must be considered in developing a sampling program for surface water or sediments including study objectives; accessibility; site topography; flow, mixing and other physical characteristics of the water body; point and diffuse sources of contamination; and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on the vertical and lateral mixing within the body of water. For sediments, dispersion depends on bottom current or flow characteristics, sediment characteristics (density, size) and geochemical properties (which effect adsorption/desorption). The sampling plan must, therefore, reflect not only the mixing characteristics of streams and lakes, but also the role of fluvial-sediment transport, deposition, and chemical sorption.

#### 5.1.1 Sampling Program Objectives

The objective of surface water sampling is to determine the surface water quality entering, leaving or remaining within the site. The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaking tanks, outfalls, etc.) or nonpoint sources (e.g., spills). The major pathways for surface water contamination (not including airborne deposition are: (a) overland runoff; (b) leachate influx to the waterbody; (c) direct waste disposal (solid or liquid) into the water body; and (d) groundwater flow influx to the water body. The relative importance of these pathways, and therefore, the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) which encompass the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (when constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the location of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc., shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly nondetectable concentrations. Such dispersion does not, however, always readily occur throughout the entire body of water; the mixing may be limited to

specific flow streams within the water body. For example, obtaining a representative sample of contamination from the center of a channel immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation are: (1) move the site far enough downstream to allow for adequate mixing, or (2) collect integrated samples in a cross section. Also, nonhomogeneous distribution is a particular problem with regard to sediment-associated contaminants which may accumulate in low-energy environments while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb on particulate matter. Nitrogen, phosphorus, and the heavy metals also may be transported by particulates. Samples will be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

The first step in selecting sampling locations; therefore, is to review site history, define hydrologic boundaries and features of the site, and identify the sources, pathways and potential distribution of contamination based on these considerations. The numbers, types and general locations of required samples upgradient, on site and downgradient can then be identified.

### 5.1.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and description of site conditions must be balanced against the costs of collection as controlled by accessibility. Wading or sampling from a stream bank often is sufficient for springs, seeps, and small streams. Bridges or piers are the first choice for locating a sampling station on a larger stream or small river; they provide ready access and also permit the sampling technician to sample any point across the stream or river. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes and reservoirs, as well as those on larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment.

If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample. Use of boats or wading to collect samples requires the use of U. S. Coast Guard approved personal flotation devices (PFDs).

Sampling in marshes or tidal areas may require the use of an all-terrain-vehicle (ATV). The same precautions mentioned above with regard to sediment disturbance will apply.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant load carried by the stream. If a gaging station is not conveniently located on a selected stream, obtaining stream flow data by direct or indirect methods shall be explored.

### 5.1.3 Frequency of Sampling

The sampling frequency and the objectives of the sampling event will be defined by the Sampling and Analysis Plan. For single-event, site- or area-characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of the contaminant between the solid and aqueous phases it may be appropriate to sample only one phase, although this often is not recommended. If samples are collected primarily for monitoring purposes, consisting of repetitive, continuing measurements to define variations and trends at a given location, water samples shall be collected at established and consistent intervals, as specified in the Sampling and Analysis Plan (often monthly or quarterly), and during droughts and floods. Samples of bottom material shall be collected from fresh deposits at least yearly, and preferably during both spring and fall seasons.

The variability in available water quality data shall be evaluated before deciding on the number and collection frequency of samples required to maintain an effective monitoring program.

## 5.2 Surface Water Sample Collection

This section presents methods for collection of samples from various surface water bodies, as well as a description of types of surface water sampling equipment. The guidance in this section should be used to develop specific sampling procedures based on site conditions and investigation goals. A summary of sampling techniques and procedures is given in Section 5.2.5.

### 5.2.1 Streams, Rivers, Outfalls and Drainage Features (Ditches, Culverts)

Methods for sampling streams, rivers, outfalls and drainage features at a single point vary from the simplest of hand sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) method.

Samples from different depths or cross-sectional locations, collected during the same sampling episode, shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be collected depend on the river's width, depth, discharge, and amount of suspended sediment. With a greater number of individual points sampled, it is more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling location can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross-section.

For larger streams greater than three feet in depth, two samples at each station shall be taken from just below the surface, and just above the bottom.

### 5.2.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify according to physical or chemical differences than rivers and streams. The relative lack of mixing requires that more samples be obtained.

The number of water sampling locations on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, the measurement of DO, pH, temperature, etc., is conducted on each aliquot of the vertical composite. In naturally-formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical grab samples shall be composited to form a single sample. These vertical samples often are collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline which is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer which is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite sample consists of several vertical samples collected at various depths.

As it is likely that poor mixing may occur in lakes with irregular shape (with bays and coves that are protected from the wind), separate composite samples may be needed to adequately represent water quality. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements now are made in-situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, dissolved oxygen, some cations and anions, and light penetration.

### 5.2.3 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type required. The most frequently used samplers are:

- Dip sampler
- Weighted bottle
- Kemmerer

The dip sampler and the weighted bottle sampler are used most often.

The criteria for selecting a sampler include:

- Disposable and/or easily decontaminated
- Inexpensive (if the item is to be disposed of)
- Ease of operation
- Nonreactive/noncontaminating - Teflon-coating, glass, stainless steel or PVC sample chambers are preferred (in that order)

Each sample (grab or each aliquot collected for compositing) shall be measured for: specific conductance; temperature; pH; and dissolved oxygen (optional) as soon as it is recovered. These analyses will provide information on water mixing/stratification and potential contamination.

5.2.3.1 Dip Sampling

Water often is sampled by filling a container, either attached to a pole or held directly, from just beneath the surface of the water (a dip or grab sample). Constituents measured in grab samples are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration that is distributed throughout the water column and in the cross section. Therefore, whenever possible it is recommended to augment dip samples with samples that represent both dissolved and suspended constituents, and both vertical and horizontal distributions. Dip sampling often is the most appropriate sampling method for springs, seeps, ditches, and small streams.

5.2.3.2 Weighted Bottle Sampling

A grab sample also can be taken using a weighted holder that allows a sample to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of a stopped glass or plastic bottle, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The procedure for sampling is as follows:

- Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
- Pull out the stopper with a sharp jerk of the sampler line.
- Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
- Raise the sampler and cap the bottle.
- Decontaminate the outside of the bottle. The bottle can be used as the sample container (as long as original bottle is an approved container).

5.2.3.3 Kemmerer

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while being lowered in a vertical position to allow free passage of water through the cylinder. A "messenger" is sent down the line when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles.

### 5.2.4 Surface Water Sampling Techniques

Most samples taken during site investigations are grab samples. Typically, surface water sampling involves immersing the sample container directly in the body of water. The following suggestions are applicable to sampling springs, seeps, ditches, culverts, small streams and other relatively small bodies of water, and are presented to help ensure that the samples obtained are representative of site conditions:

- The most representative samples will likely be collected from near mid-stream, the center of flow in a culvert, etc.

- Downstream samples shall be collected first, with subsequent samples taken while moving upstream. Care shall be taken to minimize sediment disturbance while collecting surface water samples. If necessary, sediment samples shall be collected after the corresponding surface water sample.

- Samples may be collected either by immersing the approved sample container or a glass or nalgene beaker into the water. Sample bottles (or beakers) which do not contain preservatives shall be rinsed at least once with the water to be sampled prior to sample collection.

- Care shall be taken to avoid excessive agitation of the water which may result in the loss of volatile constituents. Additionally, samples for volatile organic analyses shall be collected first, followed by the samples for other constituents.

- Measurements for temperature, pH, specific conductance, or other field parameters, as appropriate, shall be collected immediately following sample collection for laboratory analyses.

- All samples shall be handled as described in SOP F301.

- The sampling location shall be marked via wooden stake placed at the nearest bank or shore. The sampling location number shall be marked with indelible ink on the stake.

- The following information shall be recorded in the field logbook:

  - Project location, date and time.
  - Weather.
  - Sample location number and sample identification number.
  - Flow conditions (i.e., high, low, in flood, etc.) and estimate of flow rate.
  - Visual description of water (i.e., clear, cloudy, muddy, etc.).
  - On-site water quality measurements.
  - Sketch of sampling location including boundaries of water body, sample location (and depth), relative position with respect to the site, location of wood identifier stake.
  - Names of sampling personnel.
  - Sampling technique, procedure, and equipment used.

General guidelines for collection of samples from larger streams, ponds or other water bodies are as follows:

- The most representative samples are obtained from mid-channel at mid-stream depth in a well-mixed stream.
- For sampling running water, it is suggested that the farthest downstream sample be obtained first and that subsequent samples be taken as one works upstream. Work may also proceed from zones suspected of low contamination to zones of high contamination.
- It is suggested that sample containers which do not contain preservative be rinsed at least once with the water to be sampled before the sample is taken.
- To sample a pond or other standing body of water, the surface area may be divided into grids. A series of samples taken from each grid is combined into one composite sample, or several grids are selected at random.
- Care should be taken to avoid excessive agitation of the water that would result in the loss of volatile constituents.
- When obtaining samples in 40 ml septum vials for volatile organics analysis, it is important to exclude any air space in the top of the bottle and to be sure that the Teflon liner faces inward. The bottle can be turned upside down to check for air bubbles after the bottle is filled and capped.
- Do not sample at the surface unless sampling specifically for a known constituent which is immiscible and on top of the water. Instead, the sample container should be inverted, lowered to the approximate depth, and held at about a 45-degree angle with the mouth of the bottle facing upstream.
- Measurements for temperature, pH, specific conductance, or other field parameters, as appropriate shall be collected immediately following sample collection for laboratory analysis.
- All samples shall be handled as described in SOP F301.
- Items to be recorded in the Field Logbook are the same as those described above for small streams.

## 5.3 Sediment Sampling

Sediment samples usually are collected at the same locations as surface water samples. If only one sediment sample is to be collected, the sample location shall be approximately at the center of the water body. If, however, multiple samples are required, sediment samples should be collected along a cross-section to characterize the bed material. A common procedure for obtaining multiple samples is to sample at quarter points along the cross-section of flow. As with surface water samples, sediment samples should be collected from downstream to upstream.

### 5.3.1 Sampling Equipment and Techniques

A bottom-material sample may consist of a single scoop or core or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using on-shore or off-shore techniques.

When boats are used for sampling, PFDs must be provided and two individuals must undertake the sampling. An additional person shall remain on-shore in visual contact at all times.

The following samplers may be used to collect bottom materials:

- Scoop sampler
- Dredge samplers
- Bucket/hand auger
- Stainless steel spoon or trowel
- Hand-held coring instrument

#### 5.3.1.1 Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if it can be waded, the easiest and "cleanest" way to collect a sediment sample is to use a scoop sampler. This reduces the potential for cross-contamination. This method is accomplished by reaching over or wading into the water body and, while facing upstream (into the current), scooping in the sample along the bottom in the upstream direction. It is very difficult not to disturb fine-grained materials of the sediment-water interface when using this method.

#### 5.3.1.2 Dredges

Dredges are generally used to sample sediments which cannot easily be obtained using coring devices (i.e., coarse-grained or partially-cemented materials) or when large quantities of materials are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a messenger. Most dredges are heavy (up to several hundred pounds) and require use of a winch and crane assembly for sample retrieval. There are three major types of dredges: Peterson, Eckman and Ponar dredges.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends thus reducing the "shock wave" and permits direct access to the secured sample without

opening the closed jaws. The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates. Access to the secured sample through the covering screens permits subsampling of the secured material with coring tubes or Teflon scoops, thus minimizing the chance of metal contamination from the frame of the device.

5.3.1.3 Bucket (Hand) Auger

Bucket (hand) augering is a viable method for collecting sediment samples in narrow, intermittent streams or tidal flats. Typically, a 4-inch auger bucket with a cutting head is pushed and twisted into the ground and removed as the bucket is filled. The auger hole is advanced one bucket at a time, to a depth specified in the project plans.

When a specific vertical sampling interval is required, one auger bucket is used to advance the auger hole to the first desired sampling depth. If the sample at this location is to be a vertical composite of all intervals, the same bucket may be used to advance the hole, as well as collect subsequent samples in the same hole. However, if discrete grab samples are to be collected to characterize each depth, a new bucket must be placed on the end of the auger extension immediately prior to collecting the next sample. The top several inches of sediment should be removed from the bucket to minimize the changes of cross-contamination of the sample from fall-in of material from the upper portions of the hole. The bucket auger should be decontaminated between samples as outlined in SOP F502.

5.3.1.4 Stainless Steel Spoon or Trowel

For loosely packed sediments, a stainless steel scoop or trowel can be used to collect a representative sample, in narrow intermittent streams or tidal flats.

Use the scoop or trowel to collect the sample from a desired depth. Remove heavy debris, rocks, and twigs before collecting the sample. Immediately transfer the sample to the appropriate sample container. Attach a label and identification tag. Record all required information in the field logbook and on the sample log sheet, chain-of-custody record, and other required forms.

Hand-held corers are used to obtain vertically representative samples that are relatively undisturbed. They can be used in most sediments and are less intrusive that bucket augers.

The corer is prepared by placing a new eggshell catch into a new plastic liner (rinsed first with site water) and insert the liner into the decontaminated stainless steel corer. (Note: an eggshell catch may not be required if the sediment is firm enough to remain in the liner). The liner is secured in the corer with a decontaminated plastic nosecone. The flutter valve should be checked for ease of movement and to make sure it is clear of any obstructions that could prevent a tight closure. A safety line sufficiently long enough to reach the bottom and free of any frayed or worn sections should be attached to the corer.

Line up the sampler, aiming it vertically for the point where the sample will be collected. Push the core sampler in a smooth and continuous movement through the water and into the sediment until the appropriate depth is achieved. If the corer has not been completely submerged, close the flutter valve by hand and press it shut while the sample is retrieved. Warning: the flutter valve must be kept wet to seal properly. Lift the core sampler out of the water. Tilt the sampler in a horizontal manner to prevent the sample from falling out of the liner. Unscrew the nose cone. Pull the liner out of the corer. Remove the eggshell catch, if used, and

extrude the 0 to 4 inch interval of sediment into a clean aluminum pie pan. Collect the samples for volatile analysis first, using a decontaminated stainless steel spoon. Homogenize the remaining sediment and transfer it into their respective sample jars. Repeat this process until enough sediment is obtained to fill all the sample jars. Rinse the sampling equipment with site water before collecting additional sediment from that station.

If the water is shallow, the sediment samples may be collected by pushing a new liner directly into the sediment, without inserting it into the corer. Place your hand (with a clean glove) over the opening of the liner and remove the liner from the sediment. Extrude the 0 to 4 inch interval of sediment into a clean aluminum pie pan. Collect the samples for volatile analysis first, using a decontaminated stainless steel spoon. Homogenize the remaining sediment and transfer it into their respective sample jars. Repeat this process until enough sediment is obtained to fill all the sample jars. Rinse the liner with site water before collecting additional sediment from that station.

### 5.3.2 Sediment Sampling Procedure

The following general procedure should be used, where applicable, for sampling sediment from springs, seeps, small streams, ditches, or other similar small bodies of water. Procedures sampling larger bodies of water (i.e., rivers, lakes, estuaries, etc.) should be developed on a project-specific basis, as needed.

- Sediment samples shall be collected only after the corresponding surface water sample has been collected, if one is to be collected.
- Sediment samples shall be collected from downstream locations to upstream locations.
- Samples shall be collected by excavating a sufficient amount of bottom material using a scoop, beaker, spoon, trowel, or auger. Samples should be collected with the sampling device facing upstream and the sample collected from downstream to upstream. Care should be taken to minimize the loss of fine-grained materials from the sample.
- The sample shall be transferred to the appropriate sample containers. Sampling personnel shall use judgment in removing large plant fragments to limit bias caused by bio-organic accumulation.
- All samples shall be handled as described in SOP F301.
- The sampling location shall be marked via a wooden stake placed at the nearest bank or shore. The sample location number shall be marked on the stake with indelible ink.
- The following information shall be recorded in the Field Logbook:
  - Project location, date and time.
  - Weather.
  - Sample location number and sample identification number.
  - Flow conditions.
  - Sketch of sampling location including boundaries of water body, sample location, water depth, sample collection depth, relative position with respect to the site, location of wooden identifier stake.

- Chemical analyses to be performed.
- Description of sediment (refer to SOP F001).

## 6.0 QUALITY ASSURANCE RECORDS

The description of the sampling event in the field logbook shall serve as a quality assurance record. Other records include chain-of-custody and sample analysis request forms as discussed in SOP F302.

## 7.0 REFERENCES

1. Feltz, H. R., 1980. Significance of Bottom Material Data in Evaluating Water Quality in Contaminants and Sediments. Ann Arbor, Michigan, Ann Arbor Science Publishers, Inc., V. 1, p. 271-287.

2. Kittrell, F. W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135p.

3. U.S. EPA, 1991. Standard Operating Procedures and Quality Assurance Manual. Environmental Compliance Branch, USEPA Environmental Services Division, Athens, Georgia.

4. U.S. Geological Survey, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, USGS, Reston, Virginia.

# F106
# *TEST PIT AND TRENCH EXCAVATION*

# TEST PIT AND TRENCH EXCAVATION

## 1.0 PURPOSE

The purpose of this procedure is to provide general reference information and technical guidance on the excavation of exploratory test pits and trenches.

## 2.0 SCOPE

These procedures provide overall technical guidance and may be modified by site-specific requirements for field exploratory trenches and test pits. Conditions which would make trench excavation difficult (such as a shallow water table), dangerous (presence of explosive materials or underground utilities) or likely to cause environmental problems (such as potential rupture of buried containerized wastes), will require modifications to the procedures presented herein and may prevent implementation of the exploratory excavation program. Furthermore, the costs and difficulties in disposing of potentially hazardous materials removed from the test pits may constrain their use to areas where contamination potential is low. Consequently, the techniques described herein are most applicable in areas of low apparent contamination and where potentially explosive materials are not expected to be present.

## 3.0 DEFINITIONS

Trench - Trench means a narrow excavation (in relation to its length) made below the surface of the ground. In general, the depth is greater than the width, but the width of a trench (measured at the bottom) is not greater than 15 feet. If forms or other structures are installed or constructed in an excavation so as to reduce the dimension measured from the forms or structure to the side of the excavation to 15 feet or less (measured at the bottom of the excavation), the excavation is also considered to be a trench (definition from Federal Register, Vol. 54 No. 209, Tuesday, October 31, 1989, 29 CFR Part 1926 Occupational Safety and Health Standards - Excavations; Final Rule) (see Attachment A).

Test Pit - A test pit is a small excavation made below ground surface to characterize soil type and quality as well as determine the types of wastes buried. In general, a test pit is dug using a backhoe with dimensions measured as follows:

| | | |
|---|---|---|
| Width | - | Typically two to three backhoe buckets wide |
| Length | - | Typically five to 10 feet long |
| Depth | - | Typically to top of water table or one to two feet below base of fill material |

## 4.0 RESPONSIBILITIES

Project Manager - It is the responsibility of the Project Manager to ensure that field personnel responsible for trench and test pit excavation are familiar with these procedures. It also is the responsibility of the Project Manager to ensure that all appropriate documents (i.e., Test Pit Logs) have been completely and correctly filled out by the field inspector.

Field Team Leader - The Field Team Leader is responsible for the overall supervision of all test pit and trenching activities, and for ensuring that each test pit is properly and completely logged by the field inspector. It also is the responsibility of the Field Team Leader to ensure that all field inspectors have been briefed on these procedures.

Field Inspector – The Field Inspector is responsible for the direct supervision of test pit and trenching activities. It is the Field Inspector's responsibility to log each test pit, document subsurface conditions, complete appropriate forms, and to direct the test pit or trenching activities.

## 5.0 PROCEDURES

The procedures for test pit sizes, health and safety considerations, sampling, and backfilling are discussed in the following sections. Regulation for trench excavation, including trench sizes are given in the Tuesday October 31, 1989 edition of the Federal Register, 29 CFR Part 1926, "Occupation Safety and Health Standards – Excavations; Final Rule" (Attachment A).

### 5.1 Test Pit Sizes

Test pits and trenches permit detailed exploration of the nature and contamination of in-situ materials, and the characteristics and stratification of near surface materials. The size of the excavation will depend on:

- Purpose and extent of the exploration.
- Space limitations imposed by site conditions (i.e., proximity to buildings, utilities, etc.).
- Contaminants present and the potential for release to the environment.
- Stability of the materials being excavated.
- Capabilities and limitations of the excavating equipment.

Test pits normally have a width ranging from two to ten feet or greater, depending on the objectives of the excavation and the equipment used. Test trenches are elongated test pits, usually three- to six-feet wide and extending for any desired length.

Standard equipment (i.e., backhoe) is readily available to excavate to depths of up to about 15 feet. However, larger and deeper excavations may be required. Standard equipment can be used to excavate deeper than their nominal limits by stepping or benching the excavation.

### 5.2 Health and Safety Considerations

Care must be taken by all on-site personnel during every phase of the test pit or trench excavation operation to avoid possible chemical and physical hazards. Chemical hazards may occur from direct exposure to excavated wastes or inhalation of volatilized materials. Physical hazards include the possible collapse of the trench or test pit, possible injury through violent contact with excavation equipment, or explosion or other forceful reaction upon contact with utilities exposed drums or other wastes.

All test pit and trench excavation activities must be carefully detailed in the site-specific Health and Safety Plan which will specify all precautions to be observed relative to possible chemical or physical hazards associated with these operations. Respiratory and personal protective equipment to be worn by all on-site personnel involved in excavation operations also will be specified in this document.

At locations where access is not restricted, a safety zone shall be established around the excavation. Additionally, personnel should, NOT under any circumstances, enter the excavation. Prior written approval and procedures as specified in the Sampling and Analysis Plan and the project Health and Safety Plan, are required if entry into the excavation is to be considered. Additionally, a Site Health and Safety Officer familiar with excavations shall be on site and shall direct the entry procedures.

## 5.3 Logging and Sampling

Test pits and/or trenches shall be logged and sampled by the Field Inspector. Soils shall be classified and described in accordance with the procedures given in SOP F101. Test Pit Records (Attachment B) shall be legibly completed for all test pits. Samples shall only be collected from material in the equipment bucket, or from the pile of excavated materials. The excavation shall NOT be entered for the purpose of collecting samples.

## 5.4 Backfilling

Backfilling of trenches and test pits is a normally accepted practice to reduce immediate site hazards and minimize the potential for rainwater accumulation and subsequent contaminant migration.

After inspection and completion of the appropriate test pit logs, backfill material should be returned to the pit under the direction of the field inspector. Any hazardous and/or waste materials which are not returned to the excavation as backfill must be collected and properly disposed. If a low permeability layer is accidentally penetrated, or if a soil layer containing substantial quantities of contaminants is encountered, backfill material must consist of a soil-bentonite mix. The mix should be prepared in a proportion specified by the field inspector and should be covered by "clean" soil and graded to the original land contour. Where it is safe to do so, the backhoe bucket should be used to compact each one to two-foot layer of backfill as it is placed, to reduce settling and compaction. The test pit cover should be inspected and further regraded, if necessary after settling has occurred.

The following procedures apply to the excavation and backfilling of a typical test pit. Note that if a subcontractor is procured to perform the test pit operations, the subcontractor must provide both an equipment operator and a supervisor:

- The positions of the test pits shall be located in the field by the Field Team Leader.
- Utility clearance shall be obtained for all test pit locations prior to excavation.
- Excavation equipment shall be thoroughly decontaminated prior to and after each test pit excavation (see SOP F501 and SOP F502).
- A safety zone shall be established around the test pit location prior to initiation of excavation activities.
- Excavation shall commence by removing lifts of no more than approximately 6 to 12 inches of soil.
- The Field Inspector shall log the test pit soils and record observations on a Test Pit Record. Additionally, the test pit cross-section shall be sketched in the Field Logbook with notable features identified.
- If applicable, soil or waste samples shall be collected either from the backhoe bucket or from the pile of excavated materials following SOP F102.

Baker
Baker Environmental, Inc.

# TEST PIT RECORD

PROJECT: _____

TEST PIT NO.: _____

COORDINATES: EAST: _____ NORTH: _____

SURFACE ELEVATION: _____ WATER LEVEL: _____

WEATHER: _____ DATE: _____

REMARKS: _____

**DEFINITIONS**

HNu = Photoionization Detector Reading
OVA = Organic Vapor Analyzer Reading
**Lab. Class.** = USCS (ASTM D-2487) or AASHTO (ASTM D-3282)
**Lab. Moist.** = Moisture Content (ASTM D-2216) Dry Weight Basis

| Depth (ft.) | Samp. Type and No. | HNu or OVA (ppm) Field | HNu or OVA (ppm) Head-space | Lab Class. | Lab. Moist. (%) | Visual Description (Principal Constituents, Gradation, Color, Moisture Content, Organic Content, Plasticity, and Other Observations) | Elevation |
|---|---|---|---|---|---|---|---|
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| 20 | | | | | | | |

CONTRACTOR: _____ BAKER REP.: _____

EQUIPMENT: _____ TEST PIT NO.: _____

SHEET 1 OF 1

## 5.5 Test Pit Excavation Procedures

- Test pit depths (and water levels) may be measured using an engineers rule (six foot) or a weighted measuring tape. Depths shall be measured from the ground surface.
- Upon completion, test pits shall be immediately backfilled as described in Section 5.4.
- Test pit locations shall be marked with five wooden stakes; one at each corner and one in the center. The test pit number shall be recorded on the centrally located stake.
- If applicable, the test pit will be surveyed by a registered land surveyor or measured and referenced to nearby permanent site structures (i.e., buildings, curbs, fences, etc.).

## 6.0 QUALITY ASSURANCE RECORDS

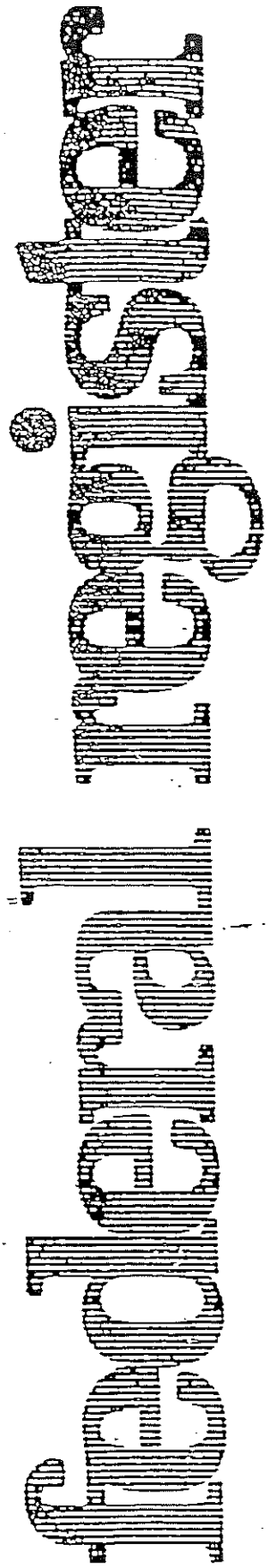
The Quality Assurance Records that should be prepared include Test Pit Records and the Field Logbook.

## 7.0 REFERENCES

OSHA , 1989. Occupational Safety and Health Standards - Excavations; Final Rule. 29 CFR Part 1926.

# ATTACHMENT A

## OSHA - EXCAVATIONS, FINAL RULE
## 29 CFR PART 1926



Tuesday
October 31, 1989

Part II

# Department of Labor

Occupational Safety and Health Administration

29 CFR Part 1926

Occupational Safety and Health Standards—Excavations; Final Rule

(ii) Installation of a support system shall be closely coordinated with the ...vation of trenches.

(f) *Sloping and benching systems.* ...oyees shall not be permitted to ... on the faces of sloped or benched excavations at levels above other employees except when employees at the lower levels are adequately protected from the hazard of falling, rolling, or sliding material or equipment.

(g) *Shield systems*—(1) *General.* (i) Shield systems shall not be subjected to loads exceeding those which the system was designed to withstand.

(ii) Shields shall be installed in a manner to restrict lateral or other hazardous movement of the shield in the event of the application of sudden lateral loads.

(iii) Employees shall be protected from the hazard of cave-ins when entering or exiting the areas protected by shields.

(iv) Employees shall not be allowed in shields when shields are being installed, removed, or moved vertically.

(2) *Additional requirement for shield systems used in trench excavations.* Excavations of earth material to a level not greater than 2 feet (.61 m) below the bottom of a shield shall be permitted, but only if the shield is designed to resist the forces calculated for the full ...pth of the trench, and there are no ...ications while the trench is open of a ...ible loss of soil from behind or ...w the bottom of the shield.

## Appendix A to Subpart P

### *Soil Classification*

(a) *Scope and application*—(1) *Scope.* This appendix describes a method of classifying soil and rock deposits based on site and environmental conditions, and on the structure and composition of the earth deposits. The appendix contains definitions, sets forth requirements; and describes acceptable visual and manual tests for use in classifying soils.

(2) *Application.* This appendix applies when a sloping or benching system is designed in accordance with the requirements set forth in § 1926.652(b)(2) as a method of protection for employees from cave-ins. This appendix also applies when timber shoring for excavations is designed as a method of protection from cave-ins in accordance with appendix C to subpart P of part 1926, and when aluminum hydraulic shoring is designed in accordance with appendix D. This Appendix also applies if other protective systems are designed and selected for use from data prepared in accordance with the requirements set forth in § 1926.652(c), and the use of the data is predicated on the use of the soil classification system set forth in this appendix.

(b) *Definitions.* The definitions and examples given below are based on, in whole ...in part, the following: American Society for Testing Materials (ASTM) Standards D653–85 and D2488: The Unified Soils Classification System, The U.S. Department of Agriculture (USDA) Textural Classification Scheme; and The National Bureau of Standards Report BSS–121.

*Cemented soil* means a soil in which the particles are held together by a chemical agent, such as calcium carbonate, such that a hand-size sample cannot be crushed into powder or individual soil particles by finger pressure.

*Cohesive soil* means clay (fine grained soil), or soil with a high clay content, which has cohesive strength. Cohesive soil does not crumble, can be excavated with vertical sideslopes, and is plastic when moist. Cohesive soil is hard to break up when dry, and exhibits significant cohesion when submerged. Cohesive soils include clayey silt, sandy clay, silty clay, clay and organic clay.

*Dry soil* means soil that does not exhibit visible signs of moisture content.

*Fissured* means a soil material that has a tendency to break along definite planes of fracture with little resistance, or a material that exhibits open cracks, such as tension cracks, in an exposed surface.

*Granular soil* means gravel, sand, or silt, (coarse grained soil) with little or no clay content. Granular soil has no cohesive strength. Some moist granular soils exhibit apparent cohesion. Granular soil cannot be molded when moist and crumbles easily when dry.

*Layered system* means two or more distinctly different soil or rock types arranged in layers. Micaceous seams or weakened planes in rock or shale are considered layered.

*Moist soil* means a condition in which a soil looks and feels damp. Moist cohesive soil can easily be shaped into a ball and rolled into small diameter threads before crumbling. Moist granular soil that contains some cohesive material will exhibit signs of cohesion between particles.

*Plastic* means a property of a soil which allows the soil to be deformed or molded without cracking, or appreciable volume change.

*Saturated soil* means a soil in which the voids are filled with water. Saturation does not require flow. Saturation, or near saturation, is necessary for the proper use of instruments such as a pocket penetrometer or sheer vane.

*Soil classification system* means, for the purpose of this subpart, a method of categorizing soil and rock deposits in a hierarchy of Stable Rock, Type A, Type B, and Type C, in decreasing order of stability. The categories are determined based on an analysis of the properties and performance characteristics of the deposits and the environmental conditions of exposure.

*Stable rock* means natural solid mineral matter that can be excavated with vertical sides and remain intact while exposed.

*Submerged soil* means soil which is underwater or is free seeping.

*Type A* means cohesive soils with an unconfined compressive strength of 1.5 ton per square foot (tsf) (144 kPa) or greater. Examples of cohesive soils are: clay, silty clay, sandy clay, clay loam and, in some cases, silty clay loam and sandy clay loam. Cemented soils such as caliche and hardpan are also considered Type A. However, no soil is Type A if:

(i) The soil is fissured; or

(ii) The soil is subject to vibration from heavy traffic, pile driving, or similar effects; or

(iii) The soil has been previously disturbed; or

(iv) The soil is part of a sloped, layered system where the layers dip into the excavation on a slope of four horizontal to one vertical (4H:1V) or greater; or

(v) The material is subject to other factors that would require it to be classified as a less stable material.

*Type B* means:

(i) Cohesive soil with an unconfined compressive strength greater than 0.5 tsf (48 kPa) but less than 1.5 tsf (144 kPa); or

(ii) Granular cohesionless soils including: angular gravel (similar to crushed rock), silt, silt loam, sandy loam and, in some cases, silty clay loam and sandy clay loam.

(iii) Previously disturbed soils except those which would otherwise be classed as Type C soil.

(iv) Soil that meets the unconfined compressive strength or cementation requirements for Type A, but is fissured or subject to vibration; or

(v) Dry rock that is not stable; or

(vi) Material that is part of a sloped, layered system where the layers dip into the excavation on a slope less steep than four horizontal to one vertical (4H:1V), but only if the material would otherwise be classified as Type B.

*Type C* means:

(i) Cohesive soil with an unconfined compressive strength of 0.5 tsf (48 kPa) or less; or

(ii) Granular soils including gravel, sand, and loamy sand; or

(iii) Submerged soil or soil from which water is freely seeping; or

(iv) Submerged rock that is not stable, or

(v) Material in a sloped, layered system where the layers dip into the excavation or a slope of four horizontal to one vertical (4H:1V) or steeper.

*Unconfined compressive strength* means the load per unit area at which a soil will fail in compression. It can be determined by laboratory testing, or estimated in the field using a pocket penetrometer, by thumb penetration tests, and other methods.

*Wet soil* means soil that contains significantly more moisture than moist soil, but in such a range of values that cohesive material will slump or begin to flow when vibrated. Granular material that would exhibit cohesive properties when moist will lose those cohesive properties when wet.

(c) *Requirements*—(1) *Classification of soil and rock deposits.* Each soil and rock deposit shall be classified by a competent person as Stable Rock, Type A, Type B, or Type C in accordance with the definitions set forth in paragraph (b) of this appendix.

(2) *Basis of classification.* The classification of the deposits shall be made based on the results of at least one visual and at least one manual analysis. Such analyses

shall be conducted by a competent person using tests described in paragraph (d) below, or in other recognized methods of soil classification and testing such as those adopted by the American Society for Testing Materials, or the U.S. Department of Agriculture textural classification system.

(3) *Visual and manual analyses.* The visual and manual analyses, such as those methods being acceptable in paragraph (d) of this appendix, shall be designed and conducted to provide sufficient quantitative and qualitative information as may be necessary to identify properly the properties, factors, and conditions affecting the classification of the deposits.

(4) *Layered systems.* In a layered system, the system shall be classified in accordance with its weakest layer. However, each layer may be classified individually where a more stable layer lies under a less stable layer.

(5) *Reclassification.* If, after classifying a deposit, the properties, factors, or conditions affecting its classification change in any way, the changes shall be evaluated by a competent person. The deposit shall be reclassified as necessary to reflect the changed circumstances.

(d) *Acceptable visual and manual tests.*—(1) *Visual tests.* Visual analysis is conducted to determine qualitative information regarding the excavation site in general, the soil adjacent to the excavation, the soil forming the sides of the open excavation, and the soil taken as samples from excavated material.

(i) Observe samples of soil that are excavated and soil in the sides of the excavation. Estimate the range of particle sizes and the relative amounts of the particle sizes. Soil that is primarily composed of fine-grained material is cohesive material. Soil composed primarily of coarse-grained sand or gravel is granular material.

(ii) Observe soil as it is excavated. Soil that remains in clumps when excavated is cohesive. Soil that breaks up easily and does not stay in clumps is granular.

(iii) Observe the side of the opened excavation and the surface area adjacent to the excavation. Crack-like openings such as tension cracks could indicate fissured material. If chunks of soil spall off a vertical side, the soil could be fissured. Small spalls are evidence of moving ground and are indications of potentially hazardous situations.

(iv) Observe the area adjacent to the excavation and the excavation itself for evidence of existing utility and other underground structures, and to identify previously disturbed soil.

(v) Observe the opened side of the excavation to identify layered systems. Examine layered systems to identify if the layers slope toward the excavation. Estimate the degree of slope of the layers.

(vi) Observe the area adjacent to the excavation and the sides of the opened excavation for evidence of surface water, water seeping from the sides of the excavation, or the location of the level of the water table.

(vii) Observe the area adjacent to the excavation and the area within the excavation for sources of vibration that may affect the stability of the excavation face.

(2) *Manual tests.* Manual analysis of soil samples is conducted to determine quantitative as well as qualitative properties of soil and to provide more information in order to classify soil properly.

(i) *Plasticity.* Mold a moist or wet sample of soil into a ball and attempt to roll it into threads as thin as ⅛-inch in diameter. Cohesive material can be successfully rolled into threads without crumbling. For example, if at least a two inch (50 mm) length of ⅛-inch thread can be held on one end without tearing, the soil is cohesive.

(ii) *Dry strength.* If the soil is dry and crumbles on its own or with moderate pressure into individual grains or fine powder, it is granular (any combination of gravel, sand, or silt). If the soil is dry and falls into clumps which break up into smaller clumps, but the smaller clumps can only be broken up with difficulty, it may be clay in any combination with gravel, sand or silt. If the dry soil breaks into clumps which do not break up into small clumps and which can only be broken with difficulty, and there is no visual indication the soil is fissured, the soil may be considered unfissured.

(iii) *Thumb penetration.* The thumb penetration test can be used to estimate the unconfined compressive strength of cohesive soils. (This test is based on the thumb penetration test described in American Society for Testing and Materials (ASTM) Standard designation D2488—"Standard Recommended Practice for Description of Soils (Visual—Manual Procedure).") Type A soils with an unconfined compressive strength of 1.5 tsf can be readily indented by the thumb; however, they can be penetrated by the thumb only with very great effort. Type C soils with an unconfined compressive strength of 0.5 tsf can be easily penetrated several inches by the thumb, and can be molded by light finger pressure. This test should be conducted on an undisturbed soil sample, such as a large clump of spoil, as soon as practicable after excavation to keep to a minimum the effects of exposure to drying influences. If the excavation is later exposed to wetting influences (rain, flooding), the classification of the soil must be changed accordingly.

(iv) *Other strength tests.* Estimates of unconfined compressive strength of soils can also be obtained by use of a pocket penetrometer or by using a hand-operated shearvane.

(v) *Drying test.* The basic purpose of the drying test is to differentiate between cohesive material with fissures, unfissured cohesive material, and granular material. The procedure for the drying test involves drying a sample of soil that is approximately one inch thick (2.54 cm) and six inches (15.24 cm) in diameter until it is thoroughly dry:

(A) If the sample develops cracks as it dries, significant fissures are indicated.

(B) Samples that dry without cracking are to be broken by hand. If considerable force is necessary to break a sample, the soil has significant cohesive material content. The soil can be classified as a unfissured cohesive material and the unconfined compressive strength should be determined.

(C) If a sample breaks easily by hand, it is either a fissured cohesive material or a granular material. To distinguish between the two, pulverize the dried clumps of the sample by hand or by stepping on them. If the clumps do not pulverize easily, the material is cohesive with fissures. If they pulverize easily into very small fragments, the material is granular.

## Appendix B to Subpart P

*Sloping and Benching*

(a) *Scope and application.* This appendix contains specifications for sloping and benching when used as methods of protecting employees working in excavations from cave-ins. The requirements of this appendix apply when the design of sloping and benching protective systems is to be performed in accordance with the requirements set forth in § 1926.652(b)(2).

(b) *Definitions.*

*Actual slope* means the slope to which an excavation face is excavated.

*Distress* means that the soil is in a condition where a cave-in is imminent or is likely to occur. Distress is evidenced by such phenomena as the development of fissures in the face of or adjacent to an open excavation; the subsidence of the edge of an excavation; the slumping of material from the face or the bulging or heaving of material from the bottom of an excavation; the spalling of material from the face of an excavation; and ravelling, i.e., small amounts of material such as pebbles or little clumps of material suddenly separating from the face of an excavation and trickling or rolling down into the excavation.

*Maximum allowable slope* means the steepest incline of an excavation face that is acceptable for the most favorable site conditions as protection against cave-ins, and is expressed as the ratio of horizontal distance to vertical rise (H:V).

*Short term exposure* means a period of time less than or equal to 24 hours that an excavation is open.

(c) *Requirements*—(1) *Soil classification.* Soil and rock deposits shall be classified in accordance with appendix A to subpart P of part 1926.

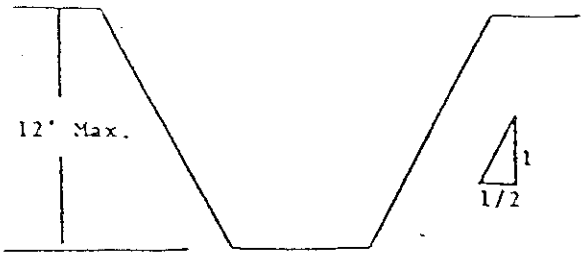
(2) *Maximum allowable slope.* The maximum allowable slope for a soil or rock deposit shall be determined from Table B–1 of this appendix.

(3) *Actual slope.* (i) The actual slope shall not be steeper than the maximum allowable slope.

(ii) The actual slope shall be less steep than the maximum allowable slope, when there are signs of distress. If that situation occurs, the slope shall be cut back to an actual slope which is at least ½ horizontal to one vertical (½H:1V) less steep than the maximum allowable slope.

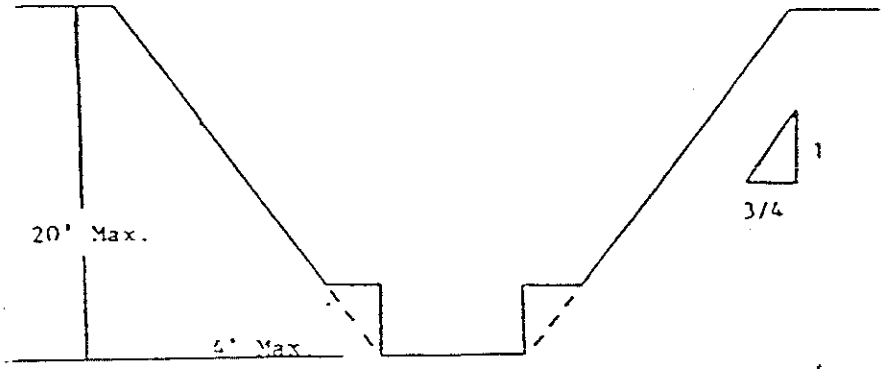
(iii) When surcharge loads from stored material or equipment, operating equipment, or traffic are present, a competent person shall determine the degree to which the actual slope must be reduced below the maximum allowable slope, and shall assure that such reduction is achieved. Surcharge loads from adjacent structures shall be evaluated in accordance with § 1926.651(i).

(4) *Configurations.* Configurations of sloping and benching systems shall be in accordance with Figure B–1.

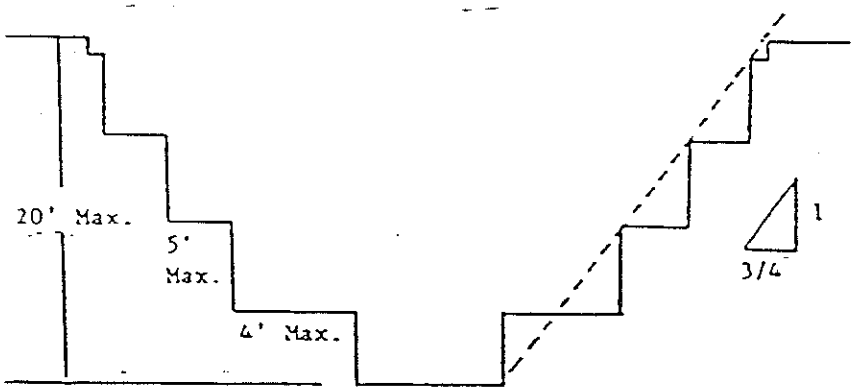



Simple Slope—Short Term

2. All benched excavations 20 feet or less in depth shall have a maximum allowable slope of ¾ to 1 and maximum bench dimensions as follows:

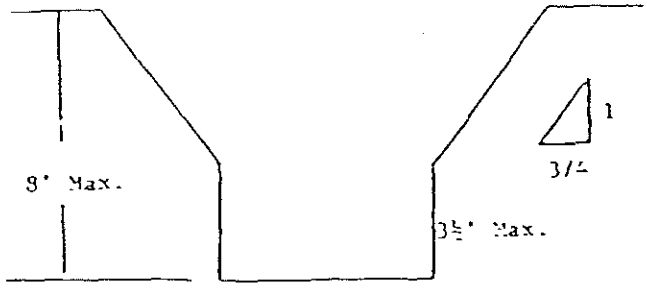



Simple Bench



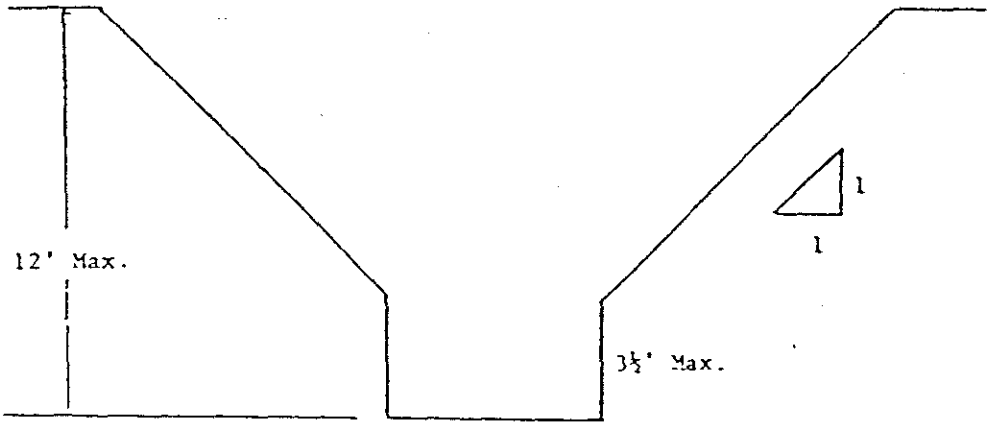

Multiple Bench

3. All excavations 8 feet or less in depth which have unsupported vertically sided lower portions shall have a maximum vertical side of 4 feet.



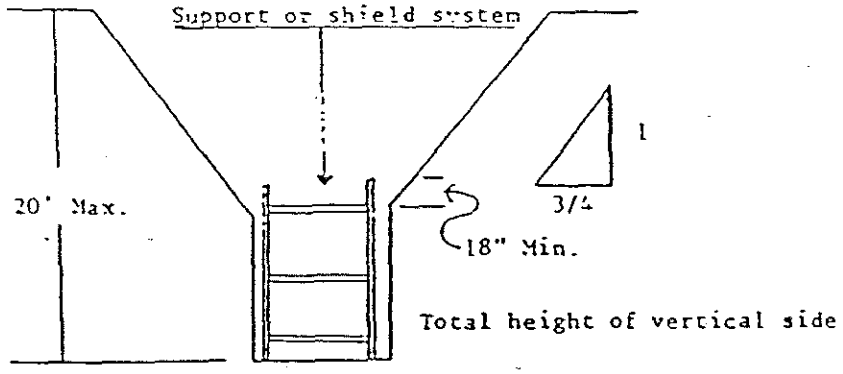

Unsupported Vertically Sided Lower Portion—Maximum 8 Feet in Depth

All excavations more than 8 feet but not more than 12 feet in depth which unsupported vertically sided lower portions shall have a maximum allowable slope of 1:1 and a maximum vertical side of 3½ feet.




Unsupported Vertically Sided Lower Portion—Maximum 12 Feet in Depth

All excavations 20 feet or less in depth which have vertically sided lower portions that are supported or shielded shall have a maximum allowable slope of ¾:1. The support or shield system must extend at least 18 inches above the top of the vertical side.

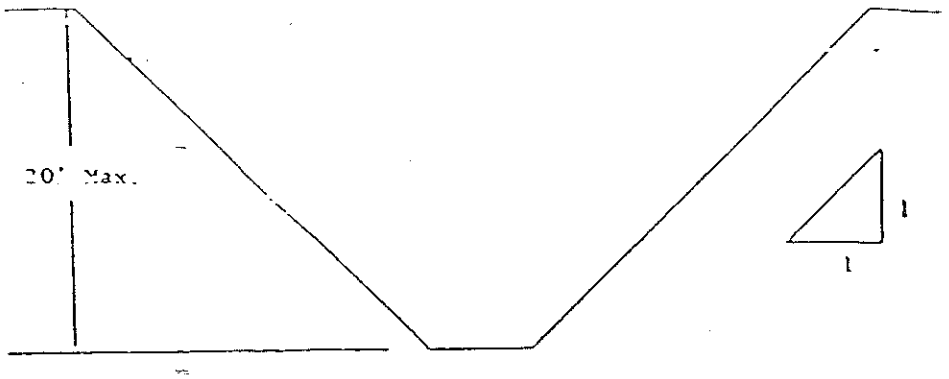



Suported or Shielded Vertically Sided Lower Portion

4. All other simple slope, compound slope, and vertically sided lower portion excavations shall be in accordance with the other options permitted under § 1926.652(b).

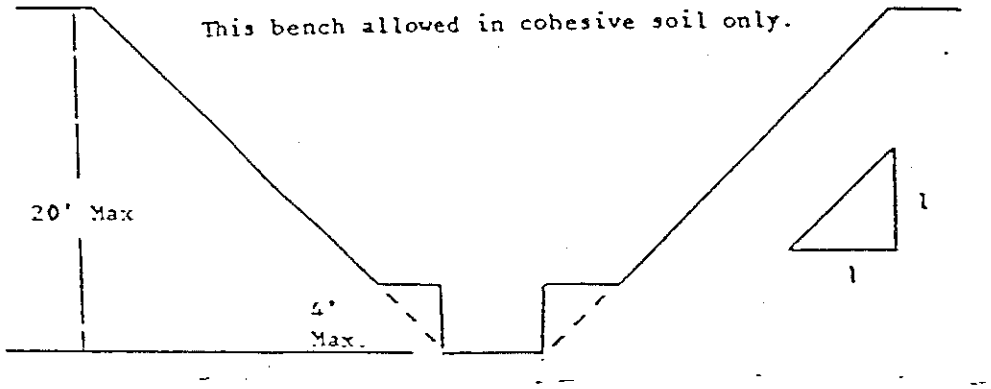
B–1.2 Excavations Made in Type B Soil

1. All simple slope excavations 20 feet or less in depth shall have a maximum allowable slope of 1:1.

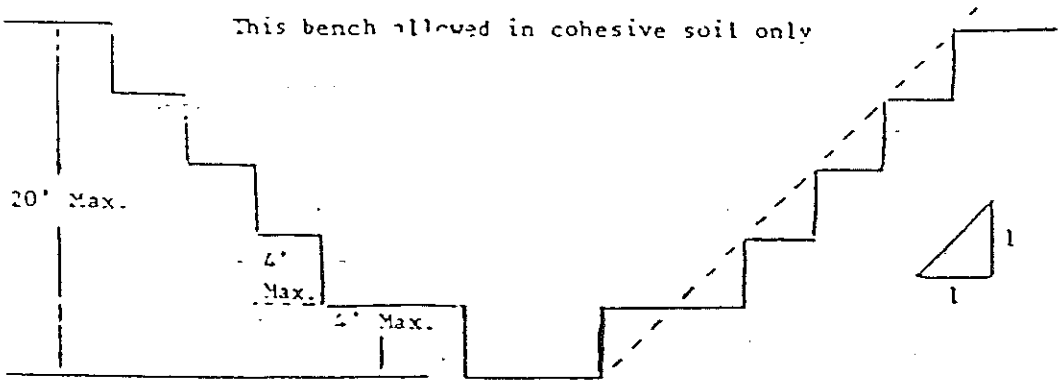



Simple Slope

2. All benched excavations 20 feet or less in depth shall have a maximum allowable slope of 1:1 and maximum bench dimensions as follows:

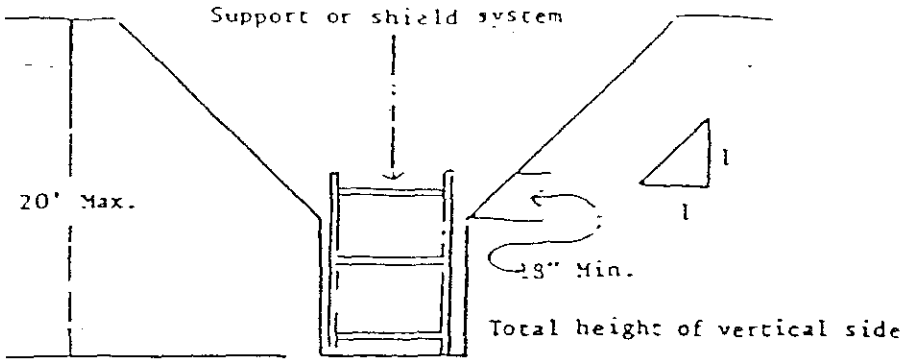



Single Bench




Multiple Bench

3. All excavations 20 feet or less in depth which have vertically sided lower portions shall be shielded or supported to a height at least 18 inches above the top of the vertical side. All such excavations shall have a maximum allowable slope of 1:1.

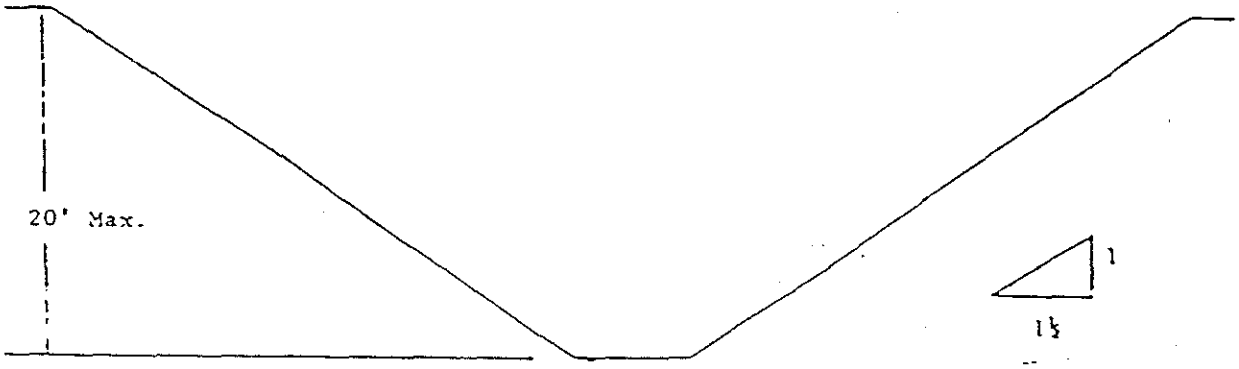



Vertically Sided Lower Portion

4. All other sloped excavations shall be in accordance with the other options permitted in § 1926.652(b).

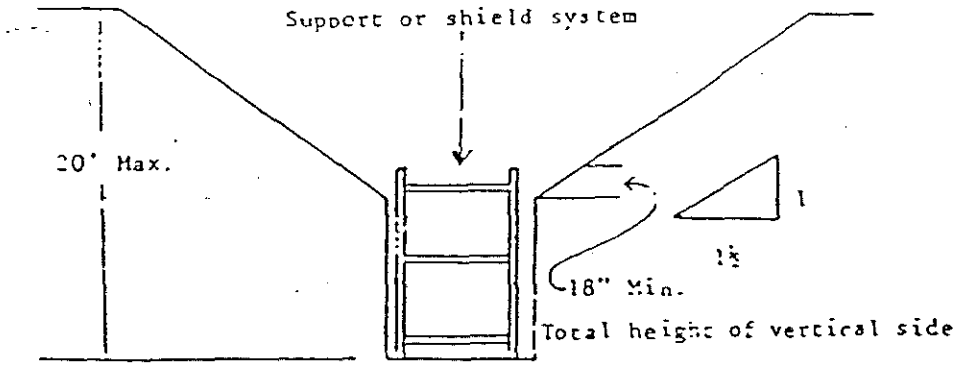
B–1.3 Excavations Made in Type C Soil

1. All simple slope excavations 20 feet or less in depth shall have a maximum allowable slope of 1½:1.




Simple Slope

2. All excavations 20 feet or less in depth which have vertically sided lower portions shall be shielded or supported to a height at least 18 inches above the top of the vertical side. All such excavations shall have a maximum allowable slope of 1½:1.

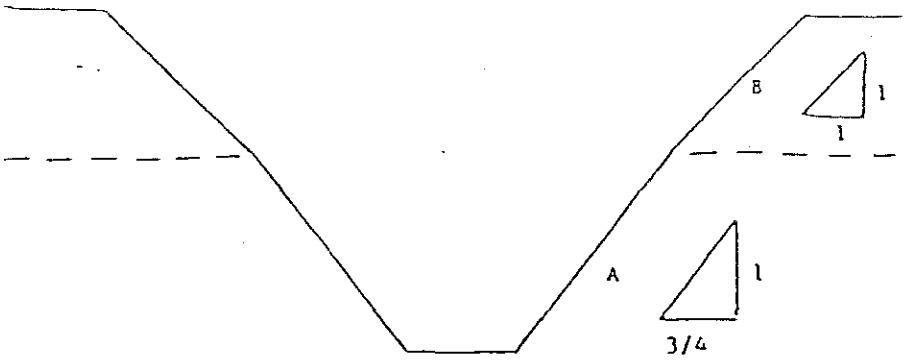



Vertical Sided Lower Portion

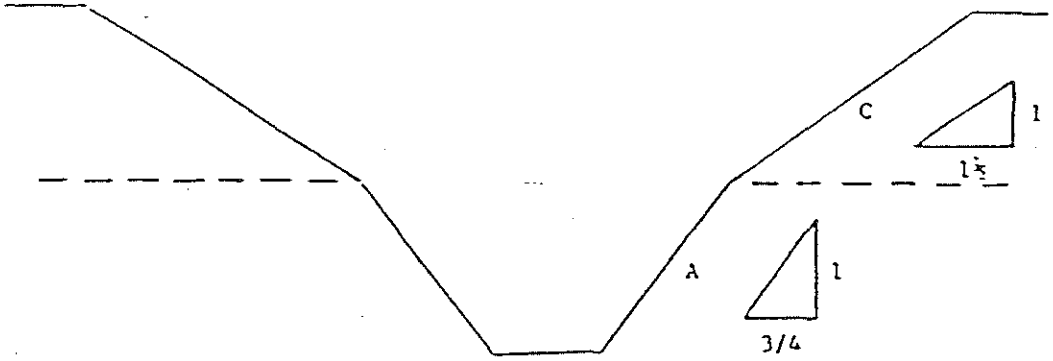
3. All other sloped excavations shall be in accordance with the other options permitted in § 1926.652(b).

B–1.4 Excavations Made in Layered Soils

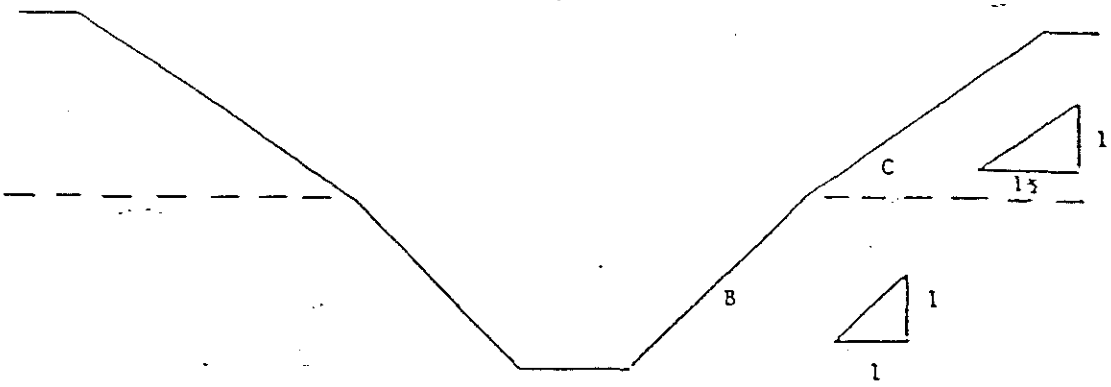
1. All excavations 20 feet or less in depth made in layered soils shall have a maximum allowable slope for each layer as set forth below.\



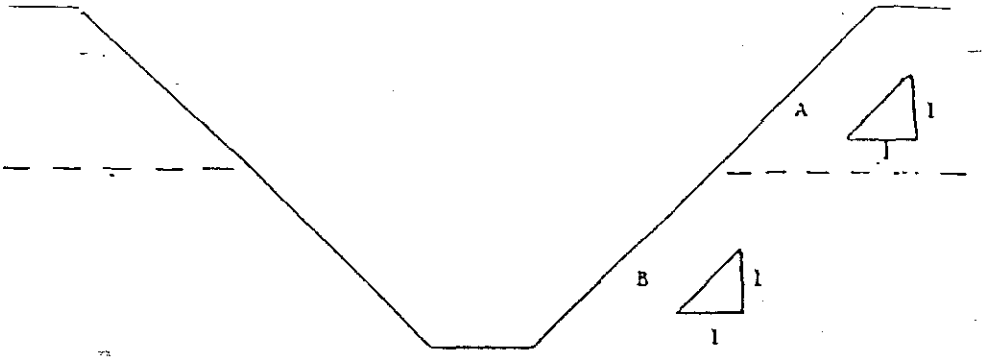

B OVER A



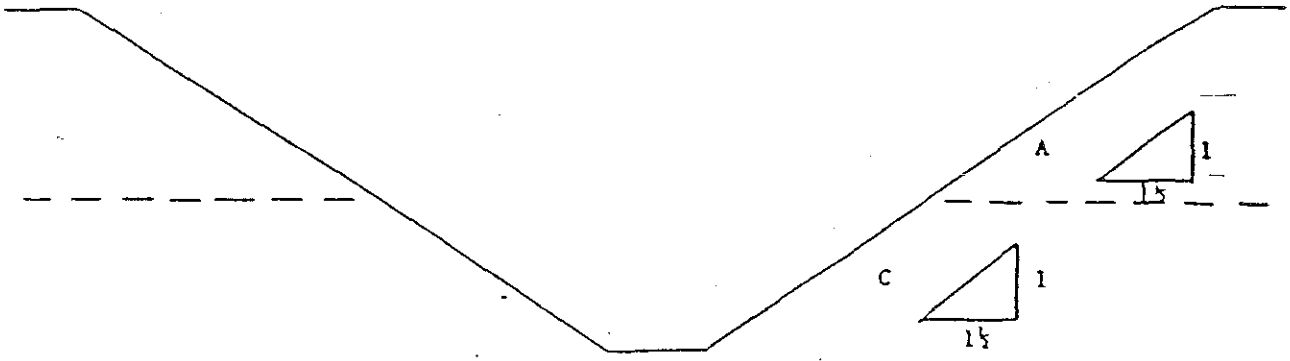

C OVER A



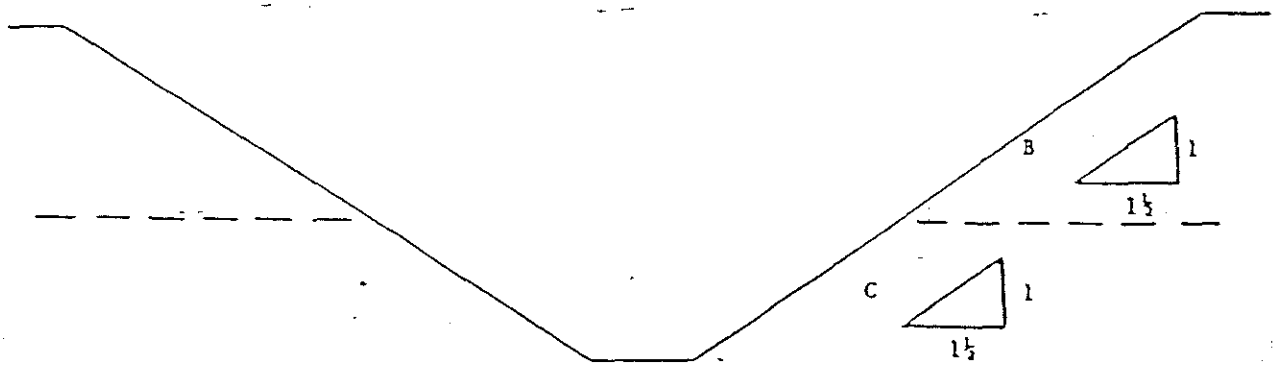

C OVER B




A OVER B




A OVER C




B OVER C

2. All other sloped excavations shall be in accordance with the other options permitted in § 1926.652(b).

## Appendix C to Subpart P

### *Timber Shoring for Trenches*

(a) *Scope.* This appendix contains information that can be used timber shoring is provided as a method of protection from cave-ins in trenches that do not exceed 20 feet (6.1 m) in depth. This appendix must be used when design of timber shoring protective systems is to be performed in accordance with § 1926.652(c)(1). Other timber shoring configurations; other systems of support such as hydraulic and pneumatic systems; and other protective systems such as sloping, benching, shielding, and freezing systems must be designed in accordance with the requirements set forth in § 1926.652(b) and § 1926.652(c).

(b) *Soil Classification.* In order to use the data presented in this appendix, the soil type or types in which the excavation is made must first be determined using the soil

classification method set forth in appendix A subpart P of this part.

(c) *Presentation of information.*

Information is presented in several forms as follows:

(1) Information is presented in tabular form in Tables C–1.1, C–1.2 and C–1.3, and Tables C–2.1, C–2.2 and C–2.3 following paragraph (g) of the appendix. Each table presents the minimum sizes of timber members to use in a shoring system, and each table contains data only for the particular soil type in which the excavation or portion of the excavation is made. The data are arranged to allow the user the flexibility to select from among several acceptable configurations of members based on varying the horizontal spacing of the crossbraces. Stable rock is exempt from shoring requirements and therefore, no data are presented for this condition.

(2) Information concerning the basis of the tabular data and the limitations of the data is presented in paragraph (d) of this appendix, and on the tables themselves.

(3) Information explaining the use of the tabular data is presented in paragraph (e) of this appendix.

(4) Information illustrating the use of the tabular data is presented in paragraph (f) of this appendix.

(5) Miscellaneous notations regarding Tables C–1.1 through C–1.3 and Tables C–2.1 through C–2.3 are presented in paragraph (g) of this Appendix.

(d) *Basis and limitations of the data.*—(1) *Dimensions of timber members.* (i) The sizes of the timber members listed in Tables C–1.1 through C–1.3 are taken from the National Bureau of Standards (NBS) report, "Recommended Technical Provisions for Construction Practice in Shoring and Sloping Trenches and Excavations." In addition, while the NBS did not recommend specific sizes for members, member sizes are based on an analysis of the sizes required for use by existing codes and on empirical practice.

(ii) The required dimensions of the members listed in Tables C–1.1 through C–1.3 refer to actual dimensions and not nominal dimensions of the timber. Employers wanting to use nominal size shoring are directed to Tables C–2.1 through C–2.3, or have this choice under § 1926.652(c)(3), and are referred to The Corps of Engineers, The Bureau of Reclamation or data from other acceptable sources.

(2) *Limitation of application.* (i) It is not intended that the timber shoring specification apply to every situation that may be experienced in the field. These data were developed to apply to the situations that are most commonly experienced in current trenching practice. Shoring systems for use in situations that are not covered by the data in this appendix must be designed as specified in § 1926.652(c).

(ii) When any of the following conditions are present, the members specified in the tables are not considered adequate. Either an alternate timber shoring system must be designed or another type of protective system designed in accordance with § 1926.652.

(A) When loads imposed by structures or by stored material adjacent to the trench weigh in excess of the load imposed by a 250-foot soil surcharge. The term "adjacent" as used here means the area within a horizontal distance from the edge of the trench equal to the depth of the trench.

(B) When vertical loads imposed on cross braces exceed a 240-pound gravity load distributed on a one-foot section of the center of the crossbrace.

(C) When surcharge loads are present from equipment weighing in excess of 20,000 pounds.

(D) When only the lower portion of a trench is shored and the remaining portion of the trench is sloped or benched unless: The sloped portion is sloped at an angle less steep than three horizontal to one vertical; or the members are selected from the tables for use at a depth which is determined from the top of the overall trench, and not from the toe of the sloped portion.

(e) *Use of Tables.* The members of the shoring system that are to be selected using this information are the cross braces, the uprights, and the wales, where wales are required. Minimum sizes of members are specified for use in different types of soil. There are six tables of information, two for each soil type. The soil type must first be determined in accordance with the soil classification system described in appendix A to subpart P of part 1926. Using the appropriate table, the selection of the size and spacing of the members is then made. The selection is based on the depth and width of the trench where the members are to be installed and, in most instances, the selection is also based on the horizontal spacing of the crossbraces. Instances where a choice of horizontal spacing of crossbracing is available, the horizontal spacing of the crossbraces must be chosen by the user before the size of any member can be determined. When the soil type, the width and depth of the trench, and the horizontal spacing of the crossbraces are known, the size and vertical spacing of the crossbraces, the size and vertical spacing of the wales, and the size and horizontal spacing of the uprights can be read from the appropriate table.

(f) *Examples to Illustrate the Use of Tables C–1.1 through C–1.3.*

(1) *Example 1.*

A trench dug in Type A soil is 13 feet deep and five feet wide.

From *Table C–1.1,* for acceptable arrangements of timber can be used.

*Arrangement #1*

Space 4×4 crossbraces at six feet horizontally and four feet vertically.

Wales are not required.

Space 3×6 uprights at six feet horizontally. This arrangement is commonly called "skip shoring."

*Arrangement #2*

Space 4×6 crossbraces at eight feet horizontally and four feet vertically.

Space 6×8 wales at four feet vertically.

Space 2×6 uprights at four feet horizontally.

*Arrangement #3*

Space 6×6 crossbraces at 10 feet horizontally and four feet vertically.

Space 6×10 wales at four feet vertically.

Space 2×6 uprights at five feet horizontally.

*Arrangement #4*

Space 6×6 crossbraces at 12 feet horizontally and four feet vertically.

Space 10×10 wales at four feet vertically.

Spaces 3×8 uprights at six feet horizontally.

(2) *Example 2.*

A trench dug in Type B soil in 13 feet deep and five feet wide. From Table C–1.2 three acceptable arrangements of members are listed.

*Arrangement #1.*

Space 6×6 crossbraces at six feet horizontally and five feet vertically.

Space 6×8 wales at five feet vertically.

Space 2×6 uprights at two feet horizontally.

*Arrangement #2*

Space 8×6 crossbraces at eight feet horizontally and five feet vertically.

Space 10×10 wales at five feet vertically.

Space 2×6 uprights at two feet horizontally.

*Arrangement #3*

Space 6×8 crossbraces at 10 feet horizontally and five feet vertically.

Space 10×12 wales at five feet vertically.

Space 2×6 uprights at two feet vertically.

(3) *Example 3.*

A trench dug in Type C soil is 13 feet deep and five feet wide.

From Table C–1.3 two acceptable arrangements of members can be used.

*Arrangement #1*

Space 8×8 crossbraces at six feet horizontally and five feet vertically.

Space 10×12 wales at five feet vertically.

Position 2×6 uprights as closely together as possible.

If water must be retained use special tongue and groove uprights to form tight sheeting.

*Arrangement #2*

Space 8×10 crossbraces at eight feet horizontally and five feet vertically.

Space 12×12 wales at five feet vertically.

Position 2×6 uprights in a close sheeting configuration unless water pressure must be resisted. Tight sheeting must be used where water must be retained.

(4) *Example 4.*

A trench dug in Type C soil is 20 feet deep and 11 feet wide. The size and spacing of members for the section of trench that is over 15 feet in depth is determined using Table C–1.3. Only one arrangement of members is provided.

Space 6×10 crossbraces at six feet horizontally and five feet vertically.

Space 12×12 wales at five feet vertically.

Use 3×8 tight sheeting.

Use of Tables C–2.1 through C–2.3 would follow the same procedures.

(g) *Notes for all Tables.*

1. Member sizes at spacings other than indicated are to be determined as specified in § 1926.652(c), "Design of Protective Systems."

2. When conditions are saturated or submerged use Tight Sheeting. Tight Sheeting refers to the use of specially-edged timber planks (e.g., tongue and groove) at least three inches thick, steel sheet piling, or similar construction that when driven or placed in position provide a tight wall to resist the lateral pressure of water and to prevent the loss of backfill material. Close Sheeting refers to the placement of planks side-by-side allowing as little space as possible between them.

3. All spacing indicated is measured center to center.

4. Wales to be installed with greater dimension horizontal.

5. If the vertical distance from the center of the lowest crossbrace to the bottom of the trench exceeds two and one-half feet, uprights shall be firmly embedded or a mudsill shall be used. Where uprights are embedded, the vertical distance from the center of the lowest crossbrace to the bottom of the trench shall not exceed 30 inches. When mudsills are used, the vertical distance shall not exceed 42 inches. Mudsills are wales that are installed at the toe of the trench side.

6. Trench jacks may be used in lieu of or in combination with timber crossbraces.

7. Placement of crossbraces. When the vertical spacing of crossbraces is four feet, place the top crossbrace no more than two feet below the top of the trench. When the vertical spacing of crossbraces is five feet, place the top crossbrace no more than 2.5 feet below the top of the trench.

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## TIMBER TRENCH SHORING - MINIMUM TIMBER REQUIREMENTS *

SOIL TYPE A $P_a$ = 25 X H + 72 psf (2 ft Surcharge)

| DEPTH OF TRENCH (FEET) | SIZE (ACTUAL) AND SPACING OF MEMBERS ** | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | CROSS BRACES | | | | | | WALES | | UPRIGHTS | | | | |
| | HORIZ. SPACING (FEET) | WIDTH OF TRENCH (FEET) | | | | | VERT. SPACING (FEET) | SIZE (IN) | VERT. SPACING (FEET) | MAXIMUM ALLOWABLE HORIZONTAL SPACING (FEET) | | | | |
| | | UP TO 4 | UP TO 6 | UP TO 9 | UP TO 12 | UP TO 15 | | | | CLOSE | 4 | 5 | 6 | 8 |
| 5 TO 10 | UP TO 6 | 4X4 | 4X4 | 4X6 | 6X6 | 6X6 | 4 | Not Req'd | --- | | | | 2X6 | |
| | UP TO 8 | 4X4 | 4X4 | 4X6 | 6X6 | 6X6 | 4 | Not Req'd | --- | | | | | 2X8 |
| | UP TO 10 | 4X6 | 4X6 | 4X6 | 6X6 | 6X6 | 4 | 8X8 | 4 | | | 2X6 | | |
| | UP TO 12 | 4X6 | 4X6 | 6X6 | 6X6 | 6X6 | 4 | 8X8 | 4 | | | | 2X6 | |
| 10 TO 15 | UP TO 6 | 4X4 | 4X4 | 4X6 | 6X6 | 6X6 | 4 | Not Req'd | --- | | | | 3X8 | |
| | UP TO 8 | 4X6 | 4X6 | 6X6 | 6X6 | 6X6 | 4 | 8X8 | 4 | | 2X6 | | | |
| | UP TO 10 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 4 | 8X10 | 4 | | | 2X6 | | |
| | UP TO 12 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 4 | 10X10 | 4 | | | | 3X8 | |
| 15 TO 20 | UP TO 6 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 4 | 6X8 | 4 | 3X6 | | | | |
| | UP TO 8 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 4 | 8X8 | 4 | 3X6 | | | | |
| | UP TO 10 | 8X8 | 8X8 | 8X8 | 8X8 | 8X10 | 4 | 8X10 | 4 | 3X6 | | | | |
| | UP TO 12 | 8X8 | 8X8 | 8X8 | 8X8 | 8X10 | 4 | 10X10 | 4 | 3X6 | | | | |
| OVER 20 | SEE NOTE 1 | | | | | | | | | | | | | |

* Mixed oak or equivalent with a bending strength not less than 850 psi.

** Manufactured members of equivalent strength may by substituted for wood.

TABLE C-1.2

TIMBER TRENCH SHORING -- MINIMUM TIMBER REQUIREMENTS *

SOIL TYPE B $P_a = 45 \times H + 72$ psf (2 ft. Surcharge)

| DEPTH OF TRENCH (FEET) | SIZE (ACTUAL) AND SPACING OF MEMBERS** | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | CROSS BRACES | | | | | | | WALES | | UPRIGHTS | | | | | |
| | HORIZ. SPACING (FEET) | WIDTH OF TRENCH (FEET) | | | | | VERT. SPACING (FEET) | SIZE (IN) | VERT. SPACING (FEET) | MAXIMUM ALLOWABLE HORIZONTAL SPACING (FEET) | | | | | |
| | | UP TO 4 | UP TO 6 | UP TO 9 | UP TO 12 | UP TO 15 | | | | CLOSE | 2 | 3 | | | |
| 5 TO 10 | UP TO 6 | 4X6 | 4X6 | 6X6 | 6X6 | 6X6 | 5 | 6X8 | 5 | | | 2X6 | | | |
| | UP TO 8 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 5 | 8X10 | 5 | | | 2X6 | | | |
| | UP TO 10 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 5 | 10X10 | 5 | | | 2X6 | | | |
| | See Note 1 | | | | | | | | | | | | | | |
| 10 TO 15 | UP TO 6 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 5 | 8X8 | 5 | | 2X6 | | | | |
| | UP TO 8 | 6X8 | 6X8 | 6X8 | 8X8 | 8X8 | 5 | 10X10 | 5 | | 2X6 | | | | |
| | UP TO 10 | 8X8 | 8X8 | 8X8 | 8X8 | 8X10 | 5 | 10X12 | 5 | | 2X6 | | | | |
| | See Note 1 | | | | | | | | | | | | | | |
| 15 TO 20 | UP TO 6 | 6X8 | 6X8 | 6X8 | 8X8 | 8X8 | 5 | 8X10 | 5 | 3X6 | | | | | |
| | UP TO 8 | 8X8 | 8X8 | 8X8 | 8X8 | 8X10 | 5 | 10X12 | 5 | 3X6 | | | | | |
| | UP TO 10 | 8X10 | 8X10 | 8X10 | 8X10 | 10X10 | 5 | 12X12 | 5 | 3X6 | | | | | |
| | See Note 1 | | | | | | | | | | | | | | |
| OVER 20 | SEE NOTE 1 | | | | | | | | | | | | | | |

* Mixed oak or equivalent with a bending strength not less than 850 psi.
** Manufactured members of equivalent strength may by substituted for wood.

## TIMBER TRENCH SHORING -- MINIMUM TIMBER REQUIREMENTS *

SOIL TYPE C $P_a$ = 80 X H + 72 psf (2 ft. Surcharge)

| DEPTH OF TRENCH (FEET) | SIZE (ACTUAL) AND SPACING OF MEMBERS** | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | CROSS BRACES | | | | | | | | | UPRIGHTS | | | | | |
| | HORIZ. SPACING (FEET) | WIDTH OF TRENCH (FEET) | | | | | VERT. SPACING (FEET) | SIZE (IN.) | VERT. SPACING (FEET) | MAXIMUM ALLOWABLE HORIZONTAL SPACING (FEET) (See Note 2) | | | | |
| | | UP TO 4 | UP TO 6 | UP TO 9 | UP TO 12 | UP TO 15 | | | | CLOSE | | | | |
| 5 TO 10 | UP TO 6 | 6X8 | 6X8 | 6X8 | 8X8 | 8X8 | 5 | 8X10 | 5 | 2X6 | | | | |
| | UP TO 8 | 8X8 | 8X8 | 8X8 | 8X8 | 8X10 | 5 | 10X12 | 5 | 2X6 | | | | |
| | UP TO 10 | 8X10 | 8X10 | 8X10 | 8X10 | 10X10 | 5 | 12X12 | 5 | 2X6 | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| 10 TO 15 | UP TO 6 | 8X8 | 8X8 | 8X8 | 8X8 | 8X10 | 5 | 10X12 | 5 | 2X6 | | | | |
| | UP TO 8 | 8X10 | 8X10 | 8X10 | 8X10 | 10X10 | 5 | 12X12 | 5 | 2X6 | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| 15 TO 20 | UP TO 6 | 8X10 | 8X10 | 8X10 | 8X10 | 10X10 | 5 | 12X12 | 5 | 3X6 | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| OVER 20 | SEE NOTE 1 | | | | | | | | | | | | | |

* Mixed Oak or equivalent with a bending strength not less than 850 psi.

** Manufactured members of equivalent strength may be substituted for wood.

TABLE C-2.1

TIMBER TRENCH SHORING -- MINIMUM TIMBER REQUIREMENTS *

SOIL TYPE A $P_a$ = 25 X H + 72 psf (2 ft. Surcharge)

| DEPTH OF TRENCH (FEET) | SIZE (S4S) AND SPACING OF MEMBERS ** | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | CROSS BRACES | | | | | | WALES | | UPRIGHTS | | | | | |
| | HORIZ. SPACING (FEET) | WIDTH OF TRENCH (FEET) | | | | | VERT. SPACING (FEET) | SIZE (IN.) | VERT. SPACING (FEET) | MAXIMUM ALLOWABLE HORIZONTAL SPACING (FEET) | | | | |
| | | UP TO 4 | UP TO 6 | UP TO 9 | UP TO 12 | UP TO 15 | | | | CLOSE | 4 | 5 | 6 | 8 |
| 5 TO 10 | UP TO 6 | 4X4 | 4X4 | 4X4 | 4X4 | 4X6 | 4 | Not Req'd | Not Req'd | | | | 4X6 | |
| | UP TO 8 | 4X4 | 4X4 | 4X4 | 4X6 | 4X6 | 4 | Not Req'd | Not Req'd | | | | | 4X8 |
| | UP TO 10 | 4X6 | 4X6 | 4X6 | 6X6 | 6X6 | 4 | 8X8 | 4 | | | 4X6 | | |
| | UP TO 12 | 4X6 | 4X6 | 4X6 | 6X6 | 6X6 | 4 | 8X8 | 4 | | | | 4X6 | |
| 10 TO 15 | UP TO 6 | 4X4 | 4X4 | 4X4 | 6X6 | 6X6 | 4 | Not Req'd | Not Req'd | | | | 4X10 | |
| | UP TO 8 | 4X6 | 4X6 | 4X6 | 6X6 | 6X6 | 4 | 6X8 | 4 | | 4X6 | | | |
| | UP TO 10 | 6X6 | 6X6 | 6X6 | 6X6 | 6X6 | 4 | 8X8 | 4 | | | 4X8 | | |
| | UP TO 12 | 6X6 | 6X6 | 6X6 | 6X6 | 6X6 | 4 | 8X10 | 4 | | 4X6 | | 4X10 | |
| 15 TO 20 | UP TO 6 | 6X6 | 6X6 | 6X6 | 6X6 | 6X6 | 4 | 6X8 | 4 | 3X6 | | | | |
| | UP TO 8 | 6X6 | 6X6 | 6X6 | 6X6 | 6X6 | 4 | 8X8 | 4 | 3X6 | 4X12 | | | |
| | UP TO 10 | 6X6 | 6X6 | 6X6 | 6X6 | 6X8 | 4 | 8X10 | 4 | 3X6 | | | | |
| | UP TO 12 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 4 | 8X12 | 4 | 3X6 | 4X12 | | | |
| OVER 20 | SEE NOTE 1 | | | | | | | | | | | | | |

* Douglas fir or equivalent with a bending strength not less than 1500 psi.

** Manufactured members of equivalent strength may be substituted for wood.

TABLE C-2.2

TIMBER TRENCH SHORING -- MINIMUM TIMBER REQUIREMENTS *

SOIL TYPE B $P_a$ = 45 X H + 72 psf (2 ft. Surcharge)

| DEPTH OF TRENCH (FEET) | SIZE (S4S) AND SPACING OF MEMBERS ** | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | CROSS BRACES | | | | | | WALES | | UPRIGHTS | | | | |
| | HORIZ. SPACING (FEET) | WIDTH OF TRENCH (FEET) | | | | VERT. SPACING (FEET) | SIZE (IN.) | VERT. SPACING (FEET) | MAXIMUM ALLOWABLE HORIZONTAL SPACING (FEET) | | | | |
| | | UP TO 4 | UP TO 6 | UP TO 9 | UP TO 12 | UP TO 15 | | | CLOSE | 2 | 3 | 4 | 6 |
| 5 TO 10 | UP TO 6 | 4X6 | 4X6 | 4X6 | 6X6 | 6X6 | 5 | 6X8 | 5 | | | 3X12 4X8 | | 4X12 |
| | UP TO 8 | 4X6 | 4X6 | 6X6 | 6X6 | 6X6 | 5 | 8X8 | 5 | | 3X8 | | 4X8 | |
| | UP TO 10 | 4X6 | 4X6 | 6X6 | 6X6 | 6X8 | 5 | 8X10 | 5 | | | 4X8 | | |
| | See Note 1 | | | | | | | | | | | | | |
| 10 TO 15 | UP TO 6 | 6X6 | 6X6 | 6X6 | 6X8 | 6X8 | 5 | 8X8 | 5 | 3X6 | 4X10 | | | |
| | UP TO 8 | 6X8 | 6X8 | 6X8 | 8X8 | 8X8 | 5 | 10X10 | 5 | 3X6 | 4X10 | | | |
| | UP TO 10 | 6X8 | 6X8 | 8X8 | 8X8 | 8X8 | 5 | 10X12 | 5 | 3X6 | 4X10 | | | |
| | See Note 1 | | | | | | | | | | | | | |
| 15 TO 20 | UP TO 6 | 6X8 | 6X8 | 6X8 | 6X8 | 8X8 | 5 | 8X10 | 5 | 4X6 | | | | |
| | UP TO 8 | 6X8 | 6X8 | 6X8 | 8X8 | 8X8 | 5 | 10X12 | 5 | 4X6 | | | | |
| | UP TO 10 | 8X8 | 8X8 | 8X8 | 8X8 | 8X8 | 5 | 12X12 | 5 | 4X6 | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| OVER 20 | SEE NOTE 1 | | | | | | | | | | | | | |

* Douglas fir or equivalent with a bending strength not less than 1500 psi.

** Manufactured members of equivalent strength may be substituted for wood.

TABLE C-2.3

TIMBER TRENCH SHORING -- MINIMUM TIMBER REQUIREMENTS *

SOIL TYPE C $P_a$ = 80 X H + 72 psf (2 ft. Surcharge)

| DEPTH OF TRENCH (FEET) | SIZE (S4S) AND SPACING OF MEMBERS ** | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | CROSS BRACES | | | | | | WALES | | UPRIGHTS | | | | | |
| | HORIZ. SPACING (FEET) | WIDTH OF TRENCH (FEET) | | | | | VERT. SPACING (FEET) | SIZE (IN) | VERT. SPACING (FEET) | MAXIMUM ALLOWABLE HORIZONTAL SPACING (FEET) | | | | |
| | | UP TO 4 | UP TO 6 | UP TO 9 | UP TO 12 | UP TO 15 | | | | CLOSE | | | | |
| 5 TO 10 | UP TO 6 | 6X6 | 6X6 | 6X6 | 6X6 | 8X8 | 5 | 8X8 | 5 | 3X6 | | | | |
| | UP TO 8 | 6X6 | 6X6 | 6X6 | 8X8 | 8X8 | 5 | 10X10 | 5 | 3X6 | | | | |
| | UP TO 10 | 6X6 | 6X6 | 8X8 | 8X8 | 8X8 | 5 | 10X12 | 5 | 3X6 | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| 10 TO 15 | UP TO 6 | 6X8 | 6X8 | 6X8 | 8X8 | 8X8 | 5 | 10X10 | 5 | 4X6 | | | | |
| | UP TO 8 | 8X8 | 8X8 | 8X8 | 8X8 | 8X8 | 5 | 12X12 | 5 | 4X6 | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| 15 TO 20 | UP TO 6 | 8X8 | 8X8 | 8X8 | 8X10 | 8X10 | 5 | 10X12 | 5 | 4X6 | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| | See Note 1 | | | | | | | | | | | | | |
| OVER 20 | SEE NOTE 1 | | | | | | | | | | | | | |

* Douglas fir or equivalent with a bending strength not less than 1500 psi.

** Manufactured members of equivalent strength may be substituted for wood.

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Appendix D to Subpart P

*inum Hydraulic Shoring for*
*hes*

*cope.* This appendix contains
i.. nation that can be used when aluminum hydraulic shoring is provided as a method of protection against cave-ins in trenches that do not exceed 20 feet (6.1m) in depth. This appendix must be used when design of the aluminum hydraulic protective system cannot be performed in accordance with § 1926.652(c)(2).

(b) *Soil Classification.* In order to use data presented in this appendix, the soil type or types in which the excavation is made must first be determined using the soil classification method set forth in appendix A of subpart P of part 1926.

(c) *Presentation of Information.* Information is presented in several forms as follows:

(1) Information is presented in tabular form in Tables D–1.1, D–1.2, D–1.3 and E–1.4. Each table presents the maximum vertical and horizontal spacings that may be used with various aluminum member sizes and various hydraulic cylinder sizes. Each table contains data only for the particular soil type in which the excavation or portion of the excavation is made. Tables D–1.1 and D–1.2 are for vertical shores in Types A and B soil. Tables D–1.3 and D1.4 are for horizontal waler systems in Types B and C soil.

(2) Information concerning the basis of the tabular data and the limitations of the data is presented in paragraph (d) of this appendix.

(3) Information explaining the use of the
r data is presented in paragraph (e) of
ppendix.

(4) Information illustrating the use of the
r data is presented in paragraph (f) of
this appendix.

(5) Miscellaneous notations (footnotes) regarding Table D–1.1 through D–1.4 are presented in paragraph (g) of this appendix.

(6) Figures, illustrating typical installations of hydraulic shoring, are included just prior to the Tables. The illustrations page is entitled "Aluminum Hydraulic Shoring; Typical Installations."

(d) *Basis and limitations of the data.*

(1) Vertical shore rails and horizontal wales are those that meet the Section Modulus requirements in the D–1 Tables. Aluminum material is 6061–T6 or material of equivalent strength and properties.

(2) Hydraulic cylinders specifications. (i) 2-inch cylinders shall be a minimum 2-inch inside diameter with a minimum safe working capacity of no less than 18,000 pounds axial compressive load at maximum extension. Maximum extension is to include full range of cylinder extensions as recommended by product manufacturer.

(ii) 3-inch cylinders shall be a minimum 3-inch inside diameter with a safe working capacity of not less than 30,000 pounds axial compressive load at extensions as recommended by product manufacturer.

(3) Limitation of application.

(i) It is not intended that the aluminum hydraulic specification apply to every situation that may be experienced in the
d. These data were developed to apply to
situations that are most commonly experienced in current trenching practice. Shoring systems for use in situations that are not covered by the data in this appendix must be otherwise designed as specified in § 1926.652(c).

(ii) When any of the following conditions are present, the members specified in the Tables are not considered adequate. In this case, an alternative aluminum hydraulic shoring system or other type of protective system must be designed in accordance with § 1926.652.

(A) When vertical loads imposed on cross braces exceed a 100 Pound gravity load distributed on a one foot section of the center of the hydraulic cylinder.

(B) When surcharge loads are present from equipment weighing in excess of 20,000 pounds.

(C) When only the lower portion of a trench is shored and the remaining portion of the trench is sloped or benched unless: The sloped portion is sloped at an angle less steep than three horizontal to one vertical; or the members are selected from the tables for use at a depth which is determined from the top of the overall trench, and not from the toe of the sloped portion.

(e) *Use of Tables D–1.1, D–1.2, D–1.3 and D–1.4.* The members of the shoring system that are to be selected using this information are the hydraulic cylinders, and either the vertical shores or the horizontal wales. When a waler system is used the vertical timber sheeting to be used is also selected from these tables. The Tables D–1.1 and D–1.2 for vertical shores are used in Type A and B soils that do not require sheeting. Type B soils that may require sheeting, and Type C soils that always require sheeting are found in the horizontal wale Tables D–1.3 and D–1.4. The soil type must first be determined in accordance with the soil classification system described in appendix A to subpart P of part 1926. Using the appropriate table, the selection of the size and spacing of the members is made. The selection is based on the depth and width of the trench where the members are to be installed. In these tables the vertical spacing is held constant at four feet on center. The tables show the maximum horizontal spacing of cylinders allowed for each size of wale in the waler system tables, and in the vertical shore tables, the hydraulic cylinder horizontal spacing is the same as the vertical shore spacing.

(f) *Example to Illustrate the Use of the Tables:*

(1) Example 1:

A trench dug in Type A soil is 6 feet deep and 3 feet wide. From Table D–1.1: Find vertical shores and 2 inch diameter cylinders spaced 6 feet on center (o.c.) horizontally and 4 feet on center (o.c.) vertically. (See Figures 1 & 3 for typical installations.)

(2) Example 2:

A trench is dug in Type B soil that does not require sheeting. 13 feet deep and 5 feet wide. From Table D–1.2: Find vertical shores and 2 inch diameter cylinders spaced 6.5 feet o.c. horizontally and 4 feet o.c. vertically. (See Figures 1 & 3 for typical installations.)

(3) A trench is dug in Type B soil that does not require sheeting, but does experience some minor raveling of the trench face. The trench is 16 feet deep and 9 feet wide. From Table D–1.2: Find vertical shores and 2 inch diameter cylinder (with special oversleeves as designated by footnote #2) spaced 5.5 feet o.c. horizontally and 4 feet o.c. vertically, plywood (per footnote (g)(7) to the D–1 Table) should be used behind the shores. (See Figures 2 & 3 for typical installations.)

(4) Example 4: A trench is dug in previously disturbed Type B soil, with characteristics of a Type C soil, and will require sheeting. The trench is 18 feet deep and 12 feet wide. 8 foot horizontal spacing between cylinders is desired for working space. From Table D–1.3: Find horizontal wale with a section modulus of 14.0 spaced at 4 feet o.c. vertically and 3 inch diameter cylinder spaced at 9 feet maximum o.c. horizontally. 3×12 timber sheeting is required at close spacing vertically. (See Figure 4 for typical installation.)

(5) Example 5: A trench is dug in Type C soil, 9 feet deep and 4 feet wide. Horizontal cylinder spacing in excess of 6 feet is desired for working space. From Table D–1.4: Find horizontal wale with a section modulus of 7.0 and 2 inch diameter cylinders spaced at 6.5 feet o.c. horizontally. Or, find horizontal wale with a 14.0 section modulus and 3 inch diameter cylinder spaced at 10 feet o.c. horizontally. Both wales are spaced 4 feet o.c. vertically. 3×12 timber sheeting is required at close spacing vertically. (See Figure 4 for typical installation.)

(g) *Footnotes, and general notes, for Tables D–1.1, D–1.2, D–1.3, and D–1.4.*

(1) For applications other than those listed in the tables, refer to § 1926.652(c)(2) for use of manufacturer's tabulated data. For trench depths in excess of 20 feet, refer to § 1926.652(c)(2) and § 1926.652(c)(3).

(2) 2 inch diameter cylinders, at this width, shall have structural steel tube (3.5×3.5×0.1875) oversleeves, or structural oversleeves of manufacturer's specification, extending the full, collapsed length.

(3) Hydraulic cylinders capacities. (i) 2 inch cylinders shall be a minimum 2-inch inside diameter with a safe working capacity of not less than 18,000 pounds axial compressive load at maximum extension. Maximum extension is to include full range of cylinder extensions as recommended by product manufacturer.

(ii) 3-inch cylinders shall be a minimum 3-inch inside diameter with a safe work capacity of not less than 30,000 pounds axial compressive load at maximum extension. Maximum extension is to include full range of cylinder extensions as recommended by product manufacturer.

(4) All spacing indicated is measured center to center.

(5) Vertical shoring rails shall have a minimum section modulus of 0.40 inch.

(6) When vertical shores are used, there must be a minimum of three shores spaced equally, horizontally, in a group.

(7) Plywood shall be 1.125 in. thick softwood or 0.75 inch, thick, 14 ply, arctic white birch (Finland form). Please note that plywood is not intended as a structural member, but only for prevention of local raveling (sloughing of the trench face) between shores.

(8) See appendix C for timber specifications.

(9) Wales are calculated for simple span conditions.

(10) See appendix D, item (d), for basis and limitations of the data.

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# ALUMINUM HYDRAULIC SHORING
# TYPICAL INSTALLATIONS

FIGURE NO. 1

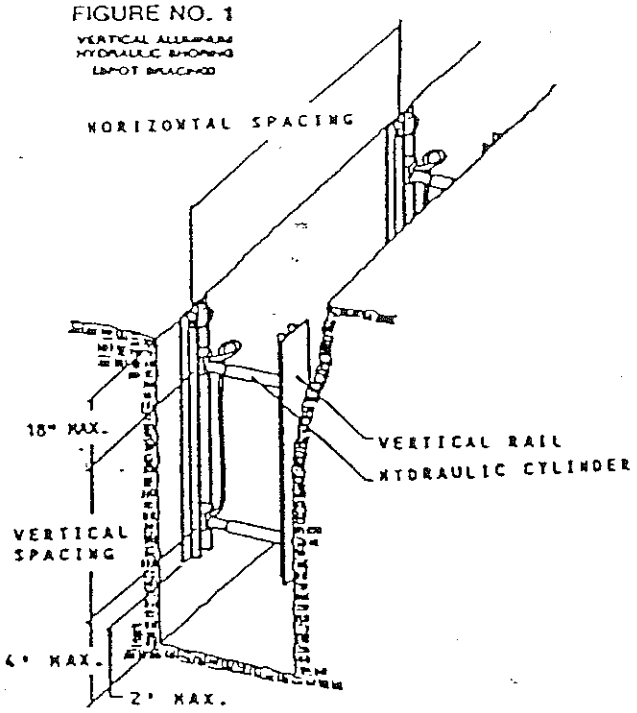



FIGURE NO. 2

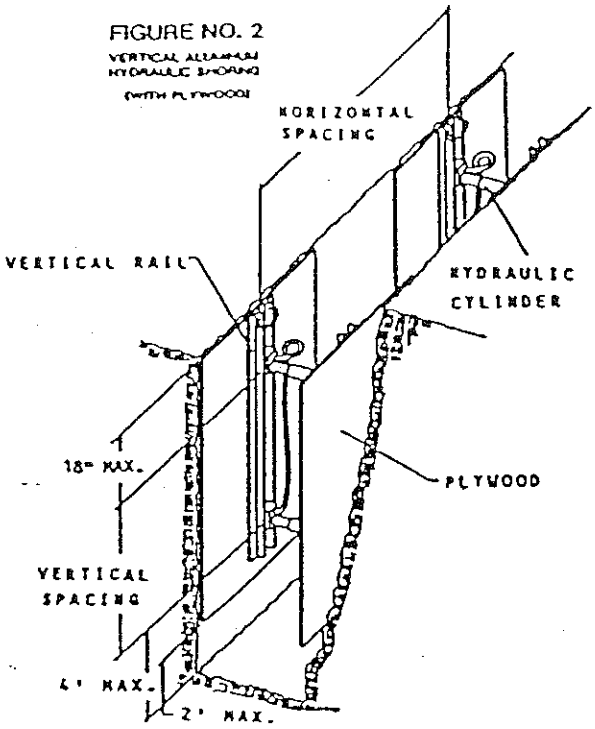



FIGURE NO. 3

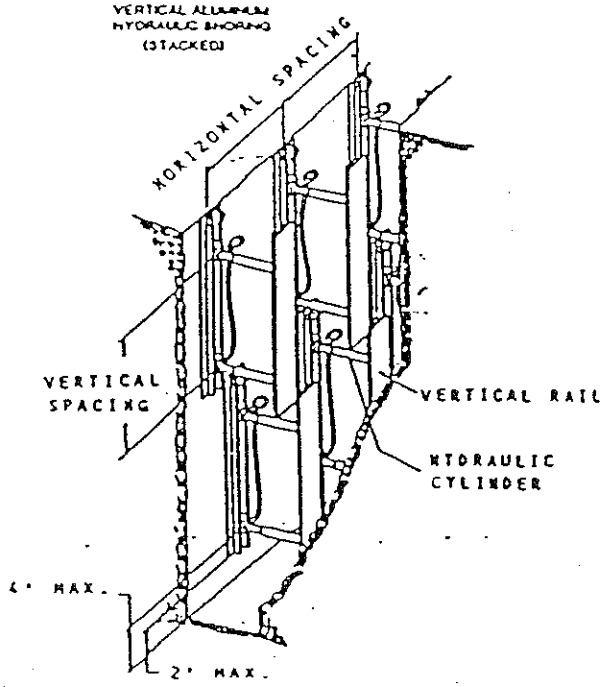



FIGURE NO. 4

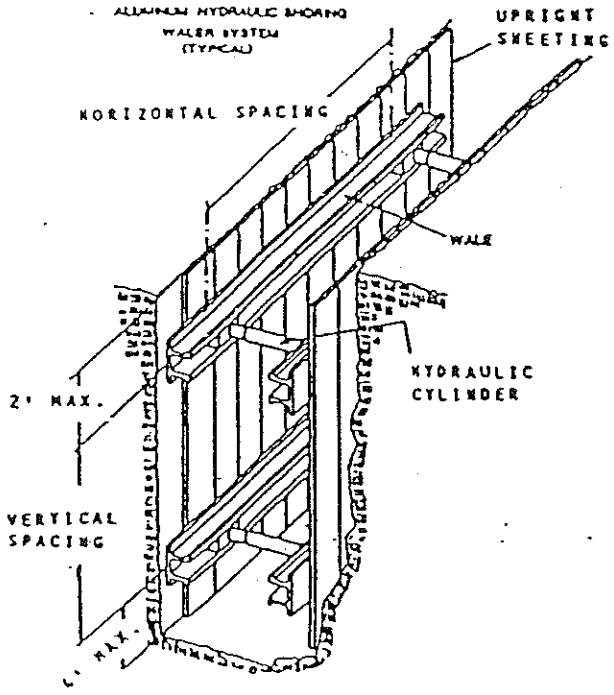


TABLE D - 1.1
ALUMINUM HYDRAULIC SHORING
VERTICAL SHORES
FOR SOIL TYPE A

| DEPTH OF TRENCH (FEET) | HYDRAULIC CYLINDERS | | | | |
|---|---|---|---|---|---|
| | MAXIMUM HORIZONTAL SPACING (FEET) | MAXIMUM VERTICAL SPACING (FEET) | WIDTH OF TRENCH (FEET) | | |
| | | | UP TO 8 | OVER 8 UP TO 12 | OVER 12 UP TO 15 |
| OVER 5 UP TO 10 | 8 | 4 | 2 INCH DIAMETER | 2 INCH DIAMETER NOTE (2) | 3 INCH DIAMETER |
| OVER 10 UP TO 15 | 8 | | | | |
| OVER 15 UP TO 20 | 7 | | | | |
| OVER 20 | NOTE (1) | | | | |

Footnotes to tables, and general notes on hydraulic shoring, are found in Appendix D, Item (g)
Note (1): See Appendix D, Item (g) (1)
Note (2): See Appendix D, Item (g) (2)

APP.ED - 1.2

ALUM... ...HYDRAULIC SHORING
VERTICAL SHORES
FOR SOIL TYPE B

| DEPTH OF TRENCH (FEET) | HYDRAULIC CYLINDERS | | | | |
|---|---|---|---|---|---|
| | MAXIMUM HORIZONTAL SPACING (FEET) | MAXIMUM VERTICAL SPACING (FEET) | WIDTH OF TRENCH (FEET) | | |
| | | | UP TO 8 | OVER 8 UP TO 12 | OVER 12 UP TO 15 |
| OVER 5 UP TO 10 | 8 | 4 | 2 INCH DIAMETER | 2 INCH DIAMETER NOTE (2) | 3 INCH DIAMETER |
| OVER 10 UP TO 15 | 6.5 | | | | |
| OVER 15 UP TO 20 | 5.5 | | | | |
| OVER 20 | NOTE (1) | | | | |

Footnotes to tables, and general notes on hydraulic shoring, are found in Appendix D, Item (g)
Note (1): See Appendix D, Item (g) (1)
Note (2): See Appendix D, Item (g) (2)

TABLE D - 1.3
ALUMINUM HYDRAULIC SHORING
WALER SYSTEMS
FOR SOIL TYPE B

| DEPTH OF TRENCH (FEET) | WALES | | HYDRAULIC CYLINDERS | | | | | | TIMBER UPRIGHTS | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | WIDTH OF TRENCH (FEET) | | | | | | MAX.HORIZ.SPACING (ON CENTER) | | |
| | | | UP TO 8 | | OVER 8 UP TO 12 | | OVER 12 UP TO15 | | | | |
| | VERTICAL SPACING (FEET) | SECTION MODULUS ($IN^3$) | HORIZ. SPACING | CYLINDER DIAMETER | HORIZ. SPACING | CYLINDER DIAMETER | HORIZ. SPACING | CYLINDER DIAMETER | SOLID SHEET | 2 FT. | 3 FT. |
| OVER 5 UP TO 10 | 4 | 3.5 | 8.0 | 2 IN | 8.0 | 2 IN NOTE(2) | 8.0 | 3 IN | — | — | 3x12 |
| | | 7.0 | 9.0 | 2 IN | 9.0 | 2 IN NOTE(2) | 9.0 | 3 IN | | | |
| | | 14.0 | 12.0 | 3 IN | 12.0 | 3 IN | 12.0 | 3 IN | | | |
| OVER 10 UP TO 15 | 4 | 3.5 | 6.0 | 2 IN | 6.0 | 2 IN NOTE(2) | 6.0 | 3 IN | — | 3x12 | — |
| | | 7.0 | 8.0 | 3 IN | 8.0 | 3 IN | 8.0 | 3 IN | | | |
| | | 14.0 | 10.0 | 3 IN | 10.0 | 3 IN | 10.0 | 3 IN | | | |
| OVER 15 UP TO 20 | 4 | 3.5 | 5.5 | 2 IN | 5.5 | 2 IN NOTE(2) | 5.5 | 3 IN | 3x12 | — | — |
| | | 7.0 | 6.0 | 3 IN | 6.0 | 3 IN | 6.0 | 3 IN | | | |
| | | 14.0 | 9.0 | 3 IN | 9.0 | 3 IN | 9.0 | 3 IN | | | |
| OVER 20 | NOTE (1) | | | | | | | | | | |

Footnotes to tables, and general notes on hydraulic shoring, are found in Appendix D, Item (g)
Notes (1): See Appendix D, item (g) (1)
Notes (2): See Appendix D, Item (g) (2)
• Consult product manufacturer and/or qualified engineer for Section Modulus of available wales.

BLE D - 1.4
ALU...NU... HYDRAULIC SHORING
WALER SYSTEMS
FOR SOIL TYPE C

| DEPTH OF TRENCH (FEET) | WALES | | HYDRAULIC CYLINDERS | | | | | | TIMBER UPRIGHTS | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | WIDTH OF TRENCH (FEET) | | | | | | MAX.HORIZ SPACING (ON CENTER) | | |
| | | | UP TO 8 | | OVER 8 UP TO 12 | | OVER 12 UP TO 15 | | | | |
| | VERTICAL SPACING (FEET) | SECTION MODULUS ($IN^3$) | HORIZ. SPACING | CYLINDER DIAMETER | HORIZ. SPACING | CYLINDER DIAMETER | HORIZ. SPACING | CYLINDER DIAMETER | SOLID SHEET | 2 FT. | 3 FT. |
| OVER 5 UP TO 10 | 4 | 3.5 | 6.0 | 2 IN | 6.0 | 2 IN NOTE(2) | 6.0 | 3 IN | 3x12 | — | — |
| | | 7.0 | 6.5 | 2 IN | 6.5 | 2 IN NOTE(2) | 6.5 | 3 IN | | | |
| | | 14.0 | 10.0 | 3 IN | 10.0 | 3 IN | 10.0 | 3 IN | | | |
| OVER 10 UP TO 15 | 4 | 3.5 | 4.0 | 2 IN | 4.0 | 2 IN NOTE(2) | 4.0 | 3 IN | 3x12 | — | — |
| | | 7.0 | 5.5 | 3 IN | 5.5 | 3 IN | 5.5 | 3 IN | | | |
| | | 14.0 | 8.0 | 3 IN | 8.0 | 3 IN | 8.0 | 3 IN | | | |
| OVER 15 UP TO 20 | 4 | 3.5 | 3.5 | 2 IN | 3.5 | 2 IN NOTE(2) | 3.5 | 3 IN | 3x12 | — | — |
| | | 7.0 | 5.0 | 3 IN | 5.0 | 3 IN | 5.0 | 3 IN | | | |
| | | 14.0 | 6.0 | 3 IN | 6.0 | 3 IN | 6.0 | 3 IN | | | |
| OVER 20 | NOTE (1) | | | | | | | | | | |

Footnotes to tables, and general notes on hydraulic shoring, are found in Appendix D, Item (g)
Notes (1): See Appendix D, item (g) (1)
Notes (2): See Appendix D, Item (g) (2)
* Consult product manufacturer and/or qualified engineer for Section Modulus of available wales.

BILLING CODE 4510-26-C

Figure 1. Aluminum Hydraulic Shoring

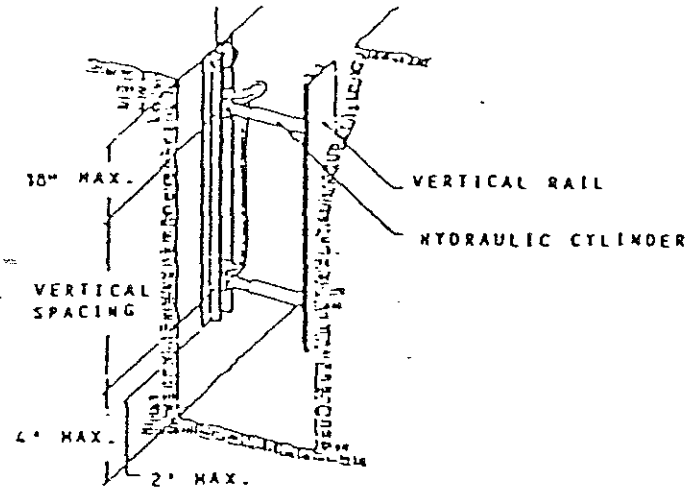
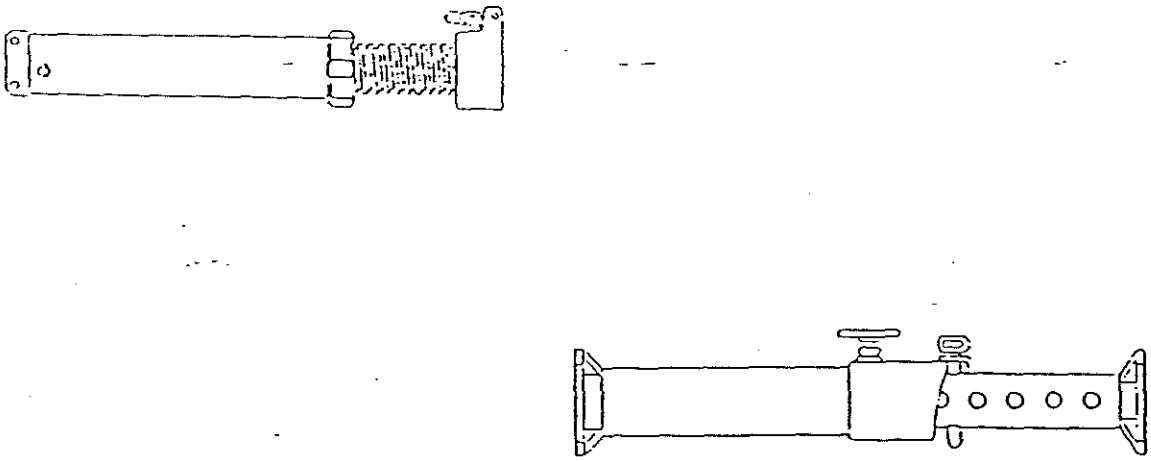



Figure 2. Pneumatic/hydraulic Shoring



BILLING CODE 4510-26-M

Figure 3. Trench Jacks (Screw Jacks)

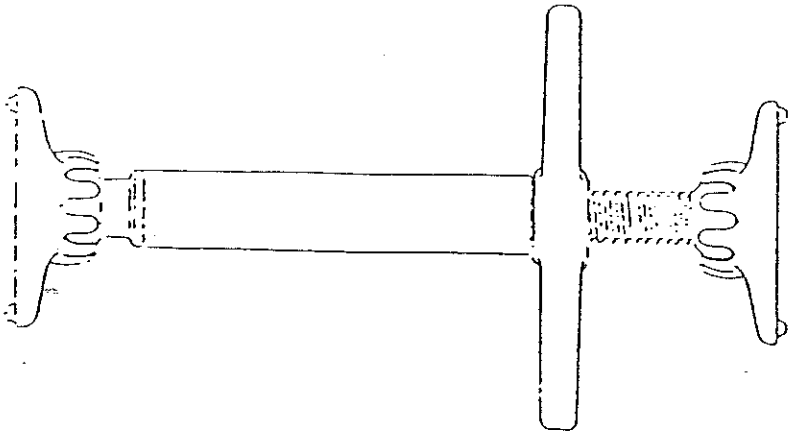
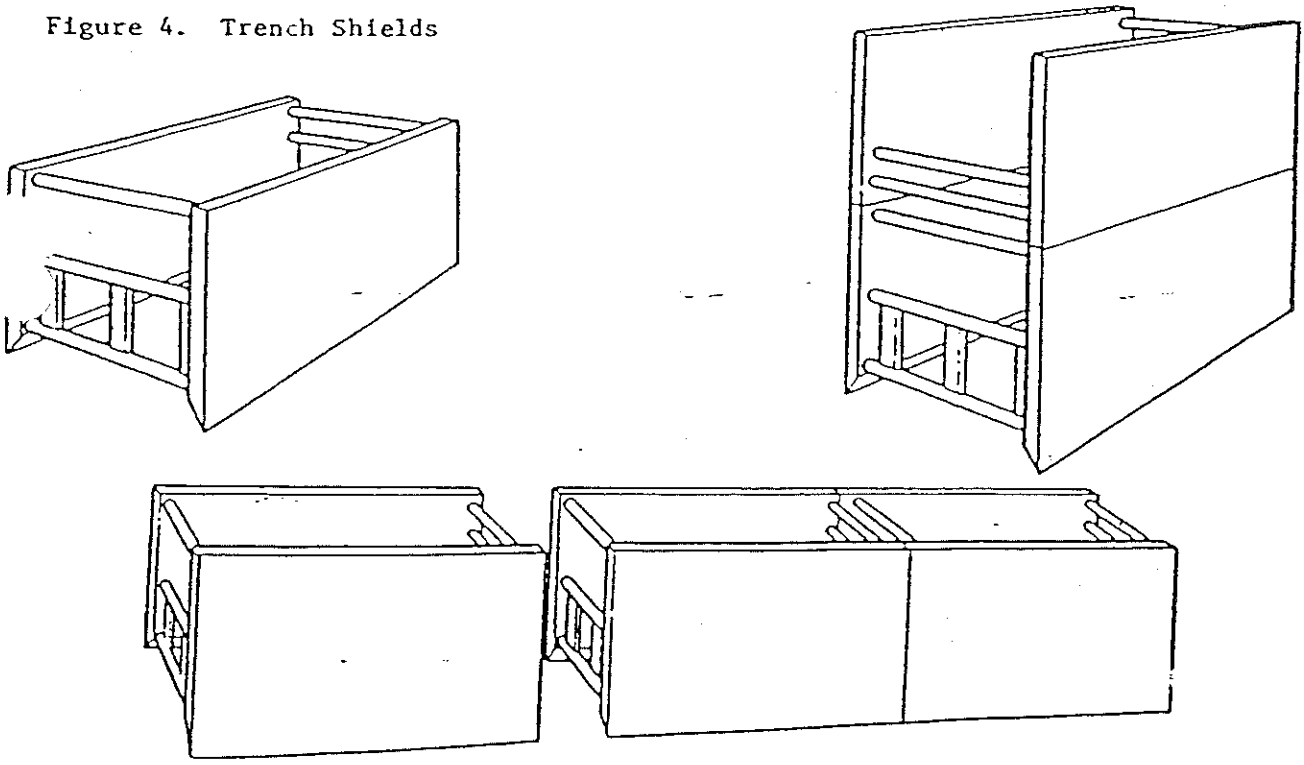


Figure 4. Trench Shields



BILLING CODE 4510-26-C

## Appendix F to Subpart P—Selection of Protective Systems

The following figures are a graphic summary of the requirements contained in subpart P for excavations 20 feet or less in depth.. Protective systems for use in excavations more than 20 feet in depth must be designed by a registered professional engineer in accordance with § 1926.652 (b) and (c).

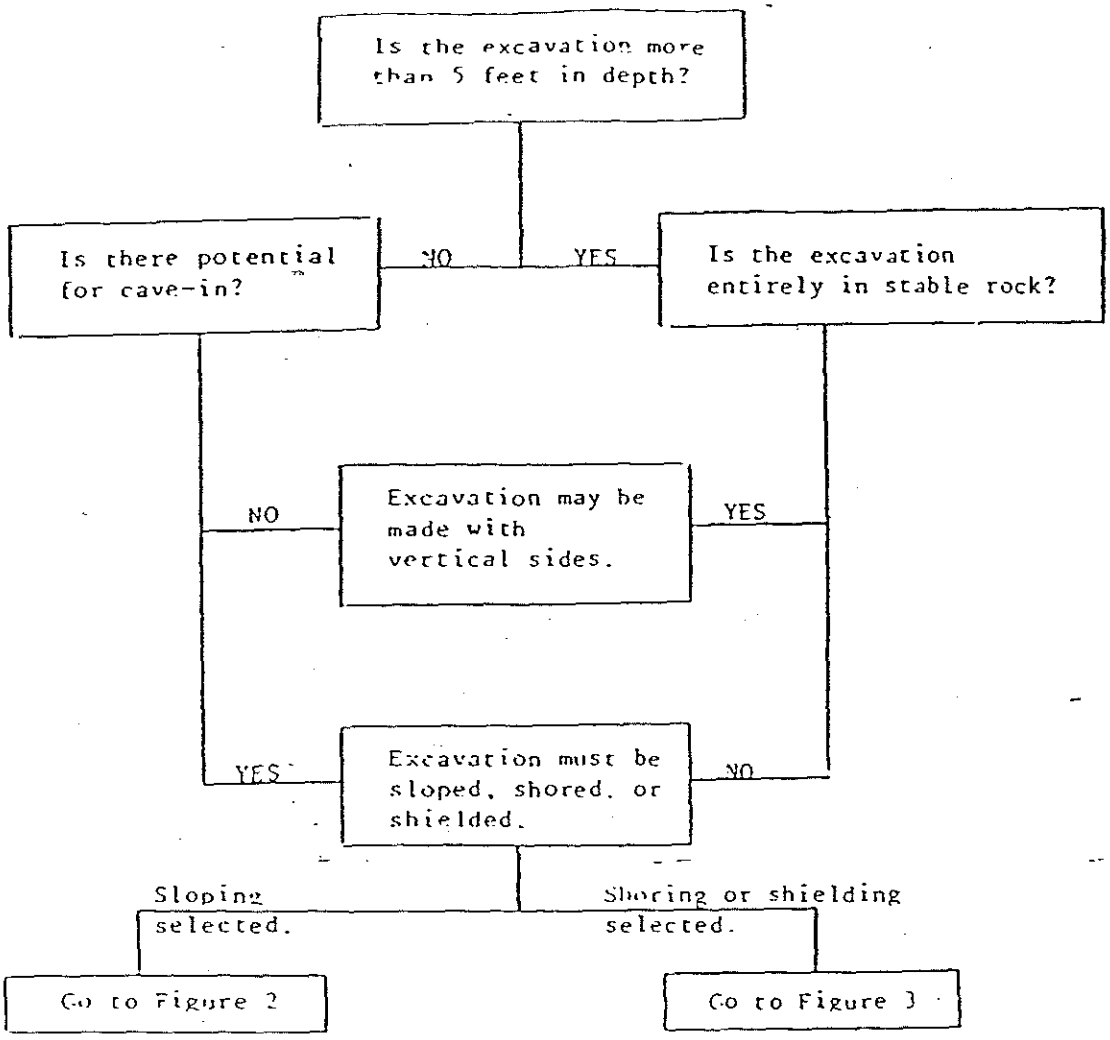



FIGURE 1 - PRELIMINARY DECISIONS

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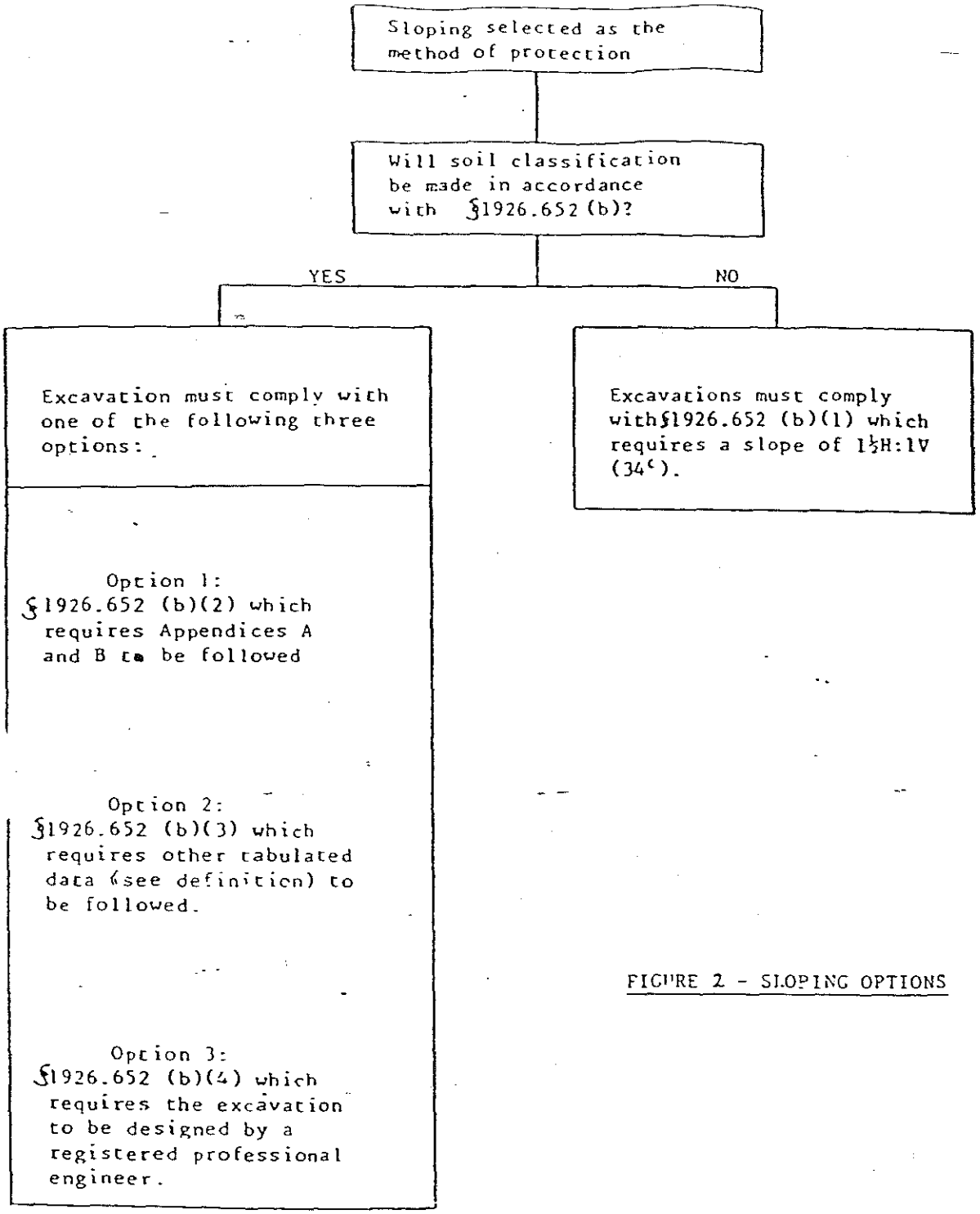
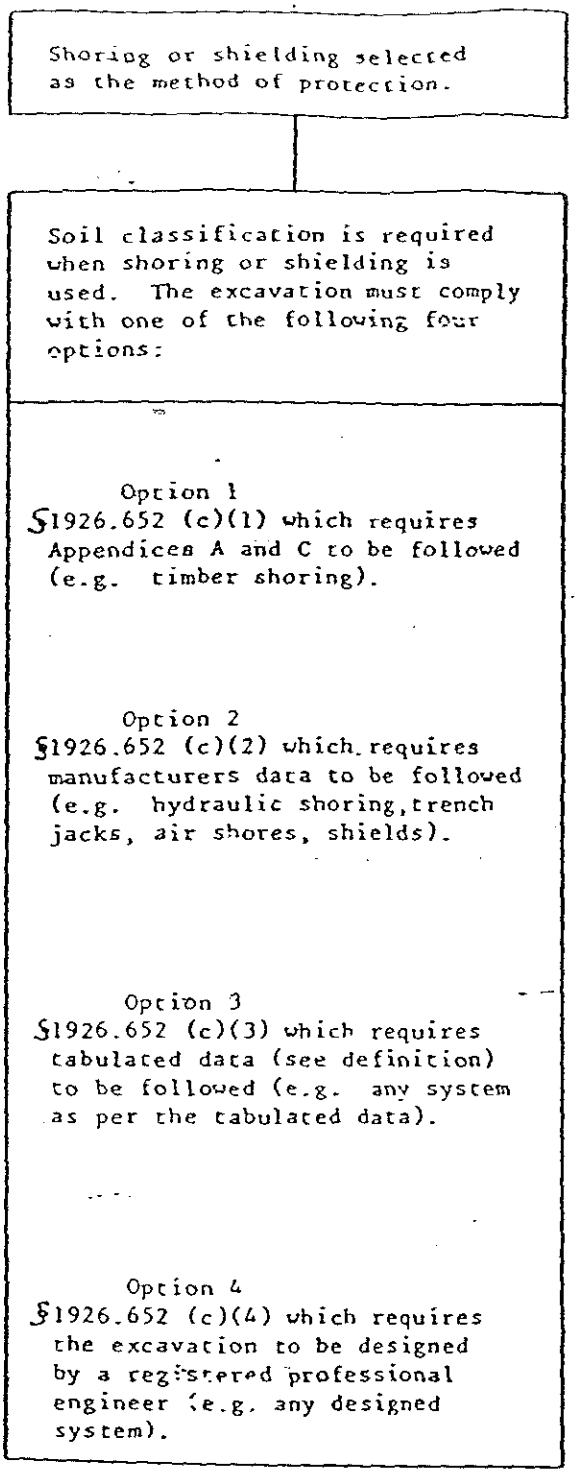



FIGURE 2 - SLOPING OPTIONS




IGURE 3 - SHORING AND SHIELDING OPTIONS

R Doc. 89-25217 Filed 10-30-89; 8:45 am]
LING CODE 4510-26-C

Baker Environmental, Inc.

Drum/Sample No.

# DRUM WASTE CHARACTERIZATION SHEET

Project Location _____ Project No. _____

Project Manager _____ Telephone _____

Logger _____ Sampler _____

Weather _____ Date _____ Time _____

Drum Type: ☐ Fiber ☐ Steel ☐ Poly ☐ Stainless Steel ☐ Nickel
☐ Poly-Lined ☐ Ring Top ☐ Closed Top ☐ Overpacked

Drum Size: 85 ☐ 55 ☐ 42 ☐ 30 ☐ 16 ☐ 10 ☐ 5 ☐ Other _____

Drum Contents: Amount Full ☐ 3/4 ☐ 1/2 ☐ 1/4 ☐ <1/4 ☐ MT ☐

Drum Condition: Good ☐ Fair ☐ Poor ☐

| | Physical State | | | | Color | Clarity | | | Layer Thickness (inches) |
|---|---|---|---|---|---|---|---|---|---|
| | Liquid | Solid | Gel | Sludge | Use Std. Colors | Clear | Cloudy | Opaque | |
| Top | | | | | | | | | |
| Middle | | | | | | | | | |
| Bottom | | | | | | | | | |

pH _____ PID _____ ppm

Rad Meter _____ mr/hr

Other _____

_____

MFG Name _____

Chemical Name _____

Additional Information: _____

_____

## LABORATORY COMPATIBILITY ANALYSES

| | Physical State | | | | Color | Clarity | | | Water Sol. | React. | pH | Hex. Sol. | Per. | Oxid. | CN | Sul. | Biel-Stein | Flash Point |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Liquid | Solid | Gel | Sludge | Use Std. Colors | Clear | Cloudy | Opaque | Sol. S or I Density | A- Air W - Water | Std. Unit | S or I | + or - | + or - | + or - | + or - | + or - | °C or °F |
| Top | | | | | | | | | | | | | | | | | | |
| Middle | | | | | | | | | | | | | | | | | | |
| Bottom | | | | | | | | | | | | | | | | | | |

Comments: _____

PCB Conc. _____ ppm Flash Point _____ °C

Data Reviewer _____ Compatibility Comp. Bulk No. _____

Field Reviewer _____

# F107
# *WASTEWATER SAMPLE ACQUISITION*

# WASTEWATER SAMPLE ACQUISITION

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information for collecting wastewater samples.

## 2.0 SCOPE

This procedure provides information for the acquisition of waste water samples. Review of the information contained herein will ensure that sample acquisition is properly conducted.

## 3.0 DEFINITIONS

Sampling Plan - A "plan of action" that guides the implementation of methods that will lead to achieving the plans objective(s).

Grab Sample - A sample that is collected at one specific sample location at a specific point in time.

Composite Sample - A representative sample made up of smaller samples collected from several different locations and/or at different points in time.

Environmental Sample - A sample of naturally occurring materials; soil, sediment, air, or water.

Waste Sample - A sample comprised of process wastes or other manmade waste material(s).

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project specific plans are in accordance with procedures where applicable, or that other approved procedures are developed. The Project Manager is responsible for development of documentation of procedures which deviate from those presented herein.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the waste water sample acquisition techniques and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the Field Investigation personnel performing the sample acquisition activities have been briefed and trained to execute these procedures.

Field Investigation Personnel - It is the responsibility of the Field Investigation Personnel to follow these procedures or to follow documented project specific procedures as outlined in the Work Plan and as directed by the Field Team Leader and Project Manager. The Field Investigation Personnel are responsible for documenting all wastewater sampling activities and ambient air monitoring results in the Field Logbook.

## 5.0 PROCEDURES

This protocol outlines procedures and equipment for the collection of representative liquid samples and sediment/sludge samples from standing lakes, ponds and lagoons, and flowing streams, rivers, channels, sewers and leachate seeps.

The collection of samples from these sources presents a unique challenge. Often sampling can be quite easy and routine (e.g., collecting a surface water sample from a two-foot deep stream). Other times, the nature of site specific conditions may dictate that: 1) special equipment is needed to access the sample, 2) appropriate health and safety measures are critical, 3) proper timing is essential due to waste release times or tidal fluctuations, and/or 4) wastewater flow rate is a factor for consideration.

Prior to sample collection, impoundment characteristics (size, depth, flow) should be recorded in the Field Logbook. Sampling should proceed from downstream locations to upstream locations so that sediment disturbance (turbidity) caused by sampling does not affect sample quality. Additionally, if a sediment sample will be collected at the same location as a liquid sample, the liquid sample must be collected first to minimize sample turbidity.

If the Sampling Plan requires that samples are to be collected from the shore of an impoundment, specific health and safety considerations must be addressed. The person collecting the sample should be fitted with a safety harness and rope secured to a sturdy, immobile object on shore. Backup personnel should be available to assist in sample collection and should be prepared and able to pull the sampler to safety if unstable banks are encountered.

To more adequately characterize the content and/or quality of an impoundment, samples may be collected away from the shoreline, often at various depths. If the content of the impoundment is suspected to be highly hazardous, the risk to sampling personnel must be weighed against the need to collect the sample. If a barge or boat is used, each person on the vessel must be equipped with a life preserver and/or lifeline.

The sampling of liquids in lakes, ponds, lagoons, streams, rivers, channels, sewers and leachate seeps is generally accomplished through the use of one of the following samplers:

- Laboratory cleaned sample bottle
- Pond sampler
- Weighted bottle sampler
- Wheaton dip sampler
- Kemmerer Depth Sampler
- Bacon Bomb Sampler

The factors that will contribute to the selection of a sampler include the width, depth and flow of the location being sampled, and whether the sample will be collected from the shore or a vessel.

For flowing liquids, tidal influence on the collected sample is an additional concern and should be addressed in the Sampling Plan. At a minimum, the stage of the tide at the time of sample collection should be recorded. Consideration should be given to sampling at varied tidal stages as well as seasonally. Tidal information can be obtained from local bait shops, newspaper listings and/or local radio or television news reports.

Samplers may encounter situations where rate of flow affects their ability to collect a sample. For fast flowing rivers and steams it may be nearly impossible to collect a mid-channel sample at a specific point. Low flowing streams and leachate seeps present the opposite problem. In these cases the sampler should attempt to locate an area where flow is obstructed and a pool is created. If this is not possible, sediment may be dug with a decontaminated trowel to create a pooled area where sufficient liquid will accumulate for sampling.

### 5.1 On-Shore

If the banks are not sloped, sampling personnel may be able to collect the liquid directly into the sample bottle. In some instances where access is limited, a pond sampler, by virtue of its extension capabilities, may be necessary. For a stream, channel or river, collect the sample at mid-depth. For standing liquid, collect the sample from just below the surface or at mid-depth. Once the sample is obtained by sample vessel, transfer it directly into the sample bottle. If volatile organic compounds (VOCs) are to be analyzed, fill the appropriate sample containers for VOCs first, then fill sample containers for other chemical analyses. Decontaminate the sampling device following procedures outlined in the Sampling Plan and/or SOP F502 before obtaining the next sample.

### 5.2 Off-Shore

Collect a liquid sample using the sample bottle or decontaminated pond sampler, if necessary. If the liquid has stratified, a sample of each strata should be collected. One of the depth samplers listed above will allow collection of discrete representative liquid samples at various depths. Proper use of the chosen sampling device includes slowly lowering and careful retrieval of the sample, immediate transfer of the liquid into the appropriate sampling container, and logbook notation of the depth at which the sample was collected. After collection, the sampling device must be decontaminated prior to obtaining the next sample.

## 6.0 QUALITY ASSURANCE RECORDS

Quality assurance records shall consist of recording sample date and acquisition time(s), sample number, sample location(s), sample depth(s), name of the Field Investigation Personnel collecting the sample(s), and Project Number in the Field Logbook. The type of container used to hold the sample and preservative agent, if needed, also will be documented, as will the method of sampling equipment decontamination. In addition, if photographs are taken of the sample site, the photograph number and direction of view shall be recorded as well.

## 7.0 REFERENCES

Field Sampling Procedures Manual. Chapter 8. New Jersey Department of Environmental Protection, Trenton, New Jersey. February 1988.

Sampling and Analysis Methods. Compilation of EPA's Sampling and Analysis Methods, USEPA, Washington, D.C. 1991.

Characterization of Hazardous Waste Sites. USEPA, Washington, D. C. 1990.

# F108
# *DRUM SAMPLING*

# DRUM SAMPLING

## 1.0 PURPOSE

The purpose of this SOP is intended to provide general information for the sampling of drums by qualified individuals in the field. Due to widely varied (and potentially hazardous) conditions posed by drum sampling, specific SOPs must be determined on a case-by-case basis. This SOP provides information to assist in ensuring that safe procedures are followed as applicable to the inspection, opening, and sampling of drums in the field.

## 2.0 SCOPE AND APPLICATION

This SOP provides technical guidance on safe and cost-effective response actions at sites containing both known and unknown drum contents. Container contents are sampled and characterized for disposal, bulking, recycling, grouping and/or classification purposes.

## 3.0 DEFINITIONS

Bung - a threaded metal or plastic plug usually positioned at the top or side of a drum.

Overpack - An external, secondary container used to enclose a packaged hazardous material, hazardous waste, or radioactive waste.

Lab Pack - a drum holding multiple individual containers of laboratory materials normally surrounded by cushioning absorbent material.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures where applicable, or that other approved procedures are developed. The Project Manager is responsible for selecting qualified individuals for the drum sampling activities.

Project Health and Safety Officer (PHSO) - The PHSO is responsible for developing a site-specific Health and Safety Plan (HASP) for drum sampling activities which include personal protection levels, air monitoring requirements, and safe drum sampling procedures.

Site Health and Safety Officer (SHSO) - The SHSO is responsible for ensuring that the proper respiratory and personal protective equipment for each member of the sampling team is selected in compliance with the HASP, and coordinating these efforts with the Field Team Leader.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the drum sampling techniques and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the Field Investigation personnel performing drum sampling activities have been briefed and trained to execute these procedures.

Field Investigation Personnel - It is the responsibility of the Field Investigation Personnel to follow these procedures or to follow documented project-specific procedures as directed by the Field Team Leader and Project Manager. The Field Investigation Personnel are responsible for documenting all sampling data on the appropriate Drum Sample Characterization Sheet presented as Attachment A and in the Field Logbook.

## 5.0 METHOD SUMMARY

Prior to sampling, drums should be inventoried and properly staged in a secure area. An inventory entails recording visual qualities of each drum and any characteristics pertinent to the contents' classification. Staging involves the organization and sometimes consolidation of drums which have similar wastes or characteristics.

## 6.0 INTERFERENCES

The practice of tapping drums to determine their contents is neither safe nor effective and should not be used if the drums are visually over pressurized (bulging) or if shock-sensitive materials are suspected. Drums that have been over pressurized, to the extent that the head is swollen several inches above the chime (beveled edge of drumtop), should not be moved. A number of devices have been developed for venting swollen drums. These devices are described in Section 7.0.

## 7.0 EQUIPMENT APPARATUS

The following are standard materials and equipment required for drum sampling:

- Health and Safety Plan
- Air monitoring equipment
- Fire extinguishing equipment
- Personal protective equipment
- Wide mouth glass jars with teflon cap liner, approximately 500 ml volume
- Uniquely numbered sample identification labels with corresponding data sheets
- One-gallon covered (paint) cans half-filled with absorbent (i.e. kitty litter or vermiculite)
- Chain-of-Custody forms
- Decontamination plan and materials
- Glass thieving tubes or Composite Liquid Waste Sampler (COLIWASA)
- Drum opening devices

### 7.1 Bung Wrench

A common method for opening drums manually is using a universal bung wrench (Figure 1, Attachment B). These wrenches have fittings made to remove nearly all commonly encountered bungs. They are usually constructed of cast-iron, brass or a bronze-beryllium, nonsparking alloy formulated to reduce the likelihood of sparks. The use of a "NONSPARKING" wrench does not completely eliminate the possibility of a spark being produced, therefore extreme caution should be exercised.

### 7.2 Drum Deheader

One means by which a drum can be opened manually (when a bung is not removable with a bung wrench) is by using a drum deheader (Figure 2, Attachment B). This tool is designed to cut the lid of a drum off (or part way off) by means of a scissors-like cutting action. This device is limited in that it can be attached only to closed head drums. Drums with removable heads must be opened by other means.

### 7.3 Backhoe Spike

The most common means used to open drums remotely for sampling is the use of a metal spike attached or welded to a backhoe bucket (Figure 3, Attachment B). In addition to being very efficient, this method can greatly reduce the likelihood of personnel exposure to the potentially hazardous nature of the drum's contents.

### 7.4 Hydraulic Drum Opener

Another remote drum opening procedure is the utilization of remotely operated hydraulic devices. One such device uses hydraulic pressure to pierce through the wall of a drum (Figure 4, Attachment B). The device consists of a manually operated pump which pressurizes oil through a length of hydraulic line.

### 7.5 Pneumatic Devices

A pneumatic bung remover consists of a compressed air supply that is controlled by a heavy-duty, two-stage regulator. A high pressure air line of desired length delivers compressed air to a pneumatic drill, which is adapted to turn a bung fitting selected to fit the bung to be removed (Figure 5, Attachment B). It should be noted that this bung removal method does not permit the slow venting of the container, and therefore appropriate precautions must be taken to reduce personnel exposure to pressurized, potentially hazardous drum contents. It also requires the container to be upright and relatively level. Bungs that are rusted shut or are in very poor condition cannot be removed with this device.

### 7.6 Tube and Spear Device

A tube and spear device is a hollow tube with a rod positioned inside the tube so that the tube acts as a guide and safety shield and the rod, which is pointed at the impact end, acts as a puncturing tool. The tube, generally a light aluminum tube (3 meters long), is positioned at the vapor space of the drum. A rigid hooking device attached to the tube goes over the chime and holds the spear securely in place. The spear is inserted in the tube and positioned against the drum wall. A sharp blow on the end of the spear drives the sharpened tip through the drum and the gas vents along the grooves. The device can be inexpensively and easily designed and constructed where needed. Once the pressure has been relieved, the bung can be removed and the drum contents sampled. For safety reasons, this device shall be operated remotely.

## 8.0 PROCEDURES

It is anticipated that the procedures for drum sampling may include a limited degree of drum handling. Therefore, it will be necessary to inspect the drum(s) for certain conditions prior to sampling.

## 8.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and which equipment and supplies will be needed.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or preclean equipment, and ensure that the equipment is in good working order.

4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site-specific Health and Safety Plan.

6. Use marking devices to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

## 8.2 Inspection

Prior to sampling, drums will be visually inspected to gain as much information as possible about their contents. Items to consider during inspection include:

- Symbols, wording, labels, or other marks indicating that drum contents are hazardous, e.g., radioactive, explosive, corrosive, toxic, or flammable.

- Symbols, wording, labels, or other marks indicating that the drum contains discarded laboratory chemicals, reagents, or other potentially dangerous materials in small-volume individual containers.

- Signs of deterioration such as corrosion, rust, and leaks.

- Signs of the chemical nature of the contents, such as residue, crystal buildup, etc. at bung opening.

- Signs that the drum is under pressure such as swelling and bulging.

- Special drum types (refer to Table 1).

- Configuration of the drumhead (ringtop or bung).

- Orientation such as whether the drum is standing upright, tilted, or lying on its side.

- Accessibility of the drum.

Monitoring will be conducted around the drums using instruments such as a gamma radiation survey instrument, organic vapor monitor (OVA or HNu), colorimetric tubes (Dräger tubes), and/or a combustible gas meter. The results can be used to classify the drums into categories such as radioactive, leaking/deteriorating, bulging, explosive/shock-sensitive, or laboratory packs.

**Personnel will not handle, move, open, sample or in anyway disturb a drum containing radioactive waste, explosive or shock-sensitive waste, laboratory packs, or biohazardous waste until specific direction and safe procedures are received from the Project Manager, PHSO and the Field Team Leader.**

When drums exhibit the characteristics of the aforementioned categories, the following procedures will be followed:

- Radioactive Wastes - If the drum exhibits radiation levels above background, normally 0.01-0.02 mrem/hr (millirem equivalent in man per hour), that are less than or equal to 2 mrem/hr, there is a possible radiation source present. Continue the investigation with caution, and inform the SHSO. If the radiation levels are greater than 2 mrem/hr there is a potential radiation hazard. Work will stop, and the Field Team Leader and Project Manager will be notified so that new procedures can be developed and implemented.

- Explosive or Shock-Sensitive Waste - If handling is necessary, exercise extreme caution, have nonessential personnel move to a safe distance, and use a grappler unit for initial handling which is constructed for explosive containment. Use nonsparking equipment and/or remote control devices.

- Bulging Drums - Do not move drums under internal pressure unless proper equipment is used, such as a grappler unit constructed for explosive containment.

- Packaged Laboratory Wastes (Lab Packs) - Lab Packs can be an ignition source for fires and sometimes contain shock-sensitive materials. Once a lab pack has been opened, a chemist or other qualified individual should visually inspect, classify and segregate the bottles, according to the hazards of the wastes. The objective of such a classification system is to ensure safe segregation of the lab packs' contents (refer to Table 2 for an example of a lab pack classification). If crystalline material is noted at the neck of any bottle, handle it as a shock-sensitive waste (due to the potential presence of picric acid, potassium permanganate or explosive mixtures resulting when the aqueous solution crystallizes), or other inimical (harmful) materials, and obtain advice from qualified personnel prior to handling.

Until drum contents are characterized, sampling personnel will assume that unlabeled drums contain hazardous materials. Personnel also should be aware that drums are frequently mislabeled and may not contain the material identified.

## 8.3 Drum Opening

Drums are to be opened and sampled in place. For opening drums manually, equipment such as a nonsparking metal (brass, bronze/manganese, aluminum, molybdenum) bung/plug wrench and a drum deheading device will be used for waste contents that are known to be nonreactive and nonexplosive, within a structurally sound drum. The drums will be grounded prior to opening either the bung or the lid

While opening drums manually with a bung wrench, the following procedures will be used:

- Drums will be positioned bung up, or, for drums with bungs on the side, laid on their sides with the bung plug up. Note that care should be taken when moving a drum into position for opening.

- Use a wrenching motion that is a slow and steady pull across the drum, using a "cheater bar" if the leverage for unscrewing the bung is poor.

- If there is evidence of incompatible chemical reactions, a sudden pressure buildup, or a release of potentially toxic fumes while the bung is being loosened, field personnel will immediately leave the area and arrange for remote drum opening equipment to be used.

- If the drum cannot be opened successfully using a nonsparking hand wrench, then other methods of drum opening (deheading or puncturing) must be considered. If deheading or puncturing a drum, it will be necessary to overpack the drum to minimize the potential for spilling the drum's contents.

- If the drum shows signs of swelling or bulging, perform all steps slowly. From a remote location, relieve excess pressure prior to drum opening using the devices listed below, if possible. If performing drum opening activities manually, place a barrier such as an explosion-resistant plastic shield between the worker and bung to deflect any gas, liquid, or solids which may be expelled as the bung is loosened.

Whenever possible, use the following remote-controlled devices for opening drums:

- A pneumatically operated impact wrench to remove drum bungs.

- A hydraulically or pneumatically operated drum piercer.

- A backhoe equipped with bronze spikes for penetrating drum tops (typical in large-scale operations).

Additional general procedures for drum opening are as follows:

- If a supplied-air respiratory protection system is used, the bank of air cylinders must be maintained outside of the work area.

- If personnel must be located near the drums being opened, place explosion-resistant plastic shields between them and the drums, in case of detonation. Locate controls for drum opening equipment, monitoring equipment, and fire suppression equipment behind the explosion-resistant plastic shield. Nonessential personnel must be positioned upwind from the drum opening and sampling operations.

- When feasible, monitor air quality continuously during drum opening, and as close as possible to the potential source of contaminants, (i.e., placing probes as close as practical without hindering drum opening operations), and hang or balance the drum opening equipment to minimize exertion.

- Do not use picks, chisels, etc. to open drums manually.

- Open exotic metal drums and polyethylene or polyvinylchloride-lined (PVC-lined) drums by removing or manually drilling the bung, while exercising extreme caution.

- Do not open or sample individual containers within laboratory packs.

- Reseal open bungs and/or drill openings as soon as possible, with new bungs or plugs to avoid explosions and/or vapor generation. If an open drum cannot be resealed, place the drum into an overpack.

- Plug any openings in pressurized drums with pressure venting caps set to a 5-psi release to allow venting of vapor pressure.

- Decontaminate and/or properly dispose of sampling equipment after each use to avoid mixing incompatible wastes and contaminating subsequent samples.

## 8.4 Drum Sampling

When sampling a previously sealed vessel, check for the presence of bottom sludge. Since some layering or stratification is likely in any solution left undisturbed over time, take a sample that represents the entire depth of the vessel.

The most widely used instrument for sampling is a glass tube commonly referred to as a glass thief (Figure 6, Attachment B). This tool is simple, cost effective, quick and collects a sample without having to decontaminate. Glass thieves are typically 6 mm to 16 mm I.D. and 48 inches long.

Drum sampling can be a very hazardous activity because it often involves direct contact with unidentified wastes. Prior to collecting any sample, field team personnel will become familiar with the procedures identified in the Sampling Plan and in this SOP.

Certain information can be construed from the drumhead configuration prior to sampling, such as:

- Removable "Whole" Lid = designed to contain solid material
- Bung opening = designed to contain liquids
- Drum Liner = may contain a highly corrosive or otherwise hazardous material

When manually sampling from a drum, use the following techniques:

- Keep sampling personnel at a safe distance while drums are being opened. Sample only after opening procedures are complete.
- Do not lean over or between other drums to reach the drum being sampled.
- Cover drum tops with plastic sheeting or other suitable uncontaminated materials to avoid excessive contact with the drum tops.
- Never stand on drums. Use mobile steps or another platform to achieve the height necessary to safely sample from the drums.
- After the drum has been opened, monitor headspace gases with no less than an explosimeter and an organic vapor analyzer. In most cases it is impossible to observe the contents of these sealed or partially sealed vessels.
- Obtain samples with either glass rods (thiefs) or with a vacuum pump and tubing. Do not use contaminated items such as discarded rags during sampling. Glass rods will be removed prior to pumping to minimize damage to pumps.
- Identify each drum with a sample number. Record the number on the Drum Waste Characterization Sheet and permanently on the drum (mark lid and side) using either a label, permanent marker, or spray paint. Cover drums with plastic sheeting and secure to minimize degradation of labeling from variable weather conditions.

8.4.1 **Procedures for using a glass thief are as follows:**

1. Remove cover from sample container.
2. Insert glass tubing almost to the bottom of the drum or until a solid layer is encountered. About one foot of tubing should extend above the drum.
3. Allow the waste in the drum to reach its natural level in the tube.
4. Cap the top of the sampling tube with a tapered stopper or thumb, ensuring liquid does not come into contact with stopper.
5. Carefully remove the capped tube from the drum and insert the uncapped end in the sample container.

6. Release stopper and allow the glass thief to drain until the container is approximately 2/3 full.

7. Remove tube from the sample container.

8. Cap the sample container tightly and place prelabeled sample container in a carrier.

9. Replace bung or lid securely on drum.

10. Break the thief into pieces inside a drum which has been designated for solid hazardous waste disposal. Previously, drum thiefs were broken and disposed inside the drum being sampled. However, this activity hindered the future disposal of liquid drum contents by introducing solid material.

11. Log all samples in the Field Logbook and on field data sheets.

12. Package samples and complete necessary paperwork.

13. Transport sample to decontamination zone in preparation for transport to analytical laboratory.

### 8.4.2 COLIWASA Sampler

The Composite Liquid Waste Sampler (COLIWASA) is designed to collect a sample from the full depth of a drum and maintain it in the transfer tube until delivery to the sample bottle. The COLIWASA (Figure 7, Attachment B) is a much cited sampler designed to permit representative sampling of multiphase wastes from drums and other containerized materials. One configuration consists of a 152 cm x 4 cm inside diameter (I.D.) section of tubing with a neoprene stopper at one end attached by a rod running the length of the tube to a locking mechanism at the other end. Manipulation of the locking mechanism opens and closes the sampler by raising and lowering the neoprene stopper.

## 9.0 QUALITY ASSURANCE/QUALITY CONTROL

The following quality assurance procedures apply:

- Document all data on standard chain-of-custody forms, field data sheets and/or within Field Logbooks.

- Operate all instrumentation in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the Sampling and Analysis Plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and must be documented in the Field Logbook.

Quality assurance records shall consist of completed Drum Waste Characterization Sheets and data entered into the Field Logbook. A sample Drum Waste Characterization Sheet is presented as Attachment A. Attachment B contains example figures of drum sampling equipment.

## 10.0 REFERENCES

Stevenson, H. L., and B. Wyman, 1991. The Facts on File Dictionary of Environmental Science. Maple-Vail Book Manufacturing Group.

NIOSH/OSHA/USCG/EPA, 1985. Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities. U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health. Publication No. 85-115.

U.S. EPA, 1986. Drum Handling Practices at Hazardous Waste Sites. Wetzel, Furman, Wickline, and Hodge, JRB Associates, McLean, Virginia. Publication No. 86-165362.

NIOSH, 1990. NIOSH Pocket Guide to Chemical Hazards. U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, Cincinnati, Ohio. Publication No. 90-117.

U.S. EPA, 1991 Compendium of ERT Waste Sampling Procedures. OSWER Directive 9360.4-07. EPA/540/P-91/008.

# ATTACHMENT A

# DRUM WASTE CHARACTERIZATION SHEET

BAKER ENVIRONMENTAL, INC.
DRUM WASTE CHARACTERIZATION SHEET

PROJECT: _____
CTO NUMBER: _____
INSPECTION DATE: _____
WEATHER: _____
BAKER REPRESENTATIVE: _____

MONITORING EQUIPMENT (Serial Number): _____
CALIBRATION DATE/TIME/INITIALS: _____
PRE-INSPECTION AIR MONITORING SCAN: _____
LEVEL OF PROTECTION: _____

| 1 | 2 | | 3 | | 4 | | 5 | 6 | | 7 | 8 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | TYPE | | CONDITION | | OVERPACKING | | | CONTENTS | | | |
| Drum Number | Size | Opening (bung or ring top) | Structural Integrity | Sealed/Exterior Contamination | Required Y/N | Fit Overpack Y/N | Head-space Reading | Volume | Phases | Sample ID Number | Sample Type (Sludge, Liquid, Sediment) |
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# ATTACHMENT B

# FIGURES

Figure 1: Universal Bung Wrench

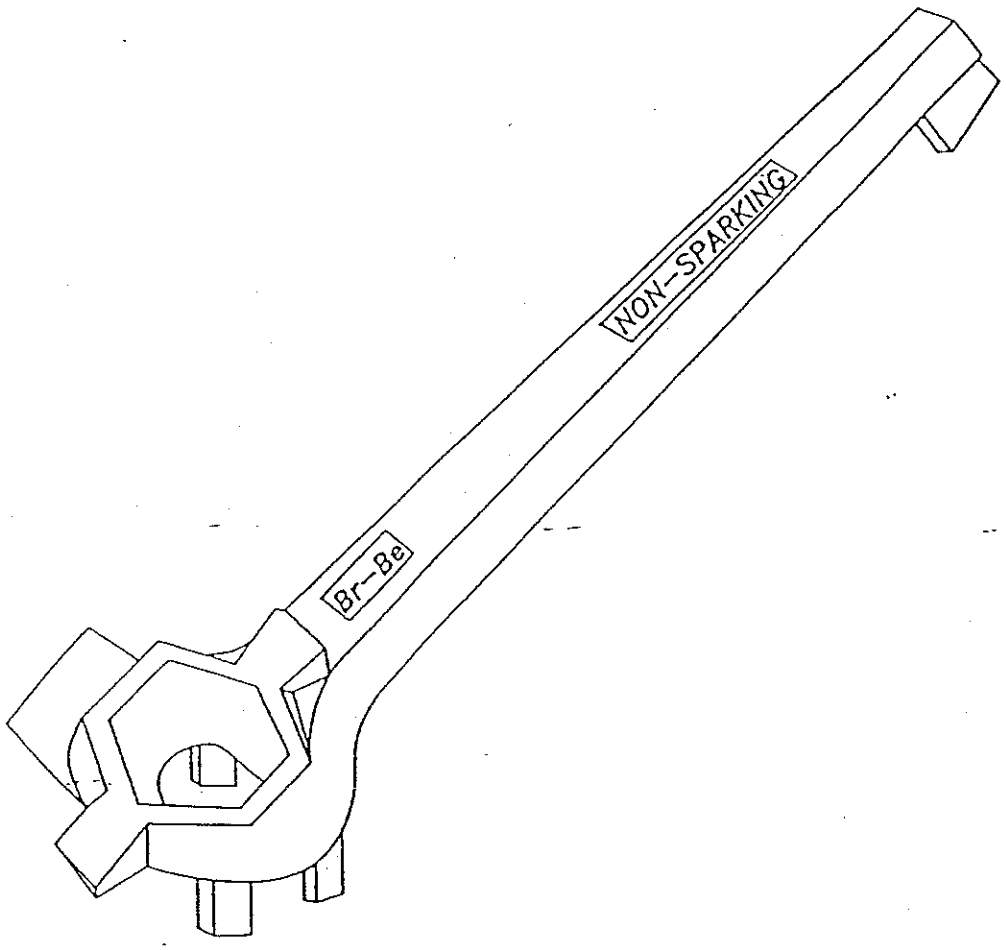


Figure 2: Drum Deheader

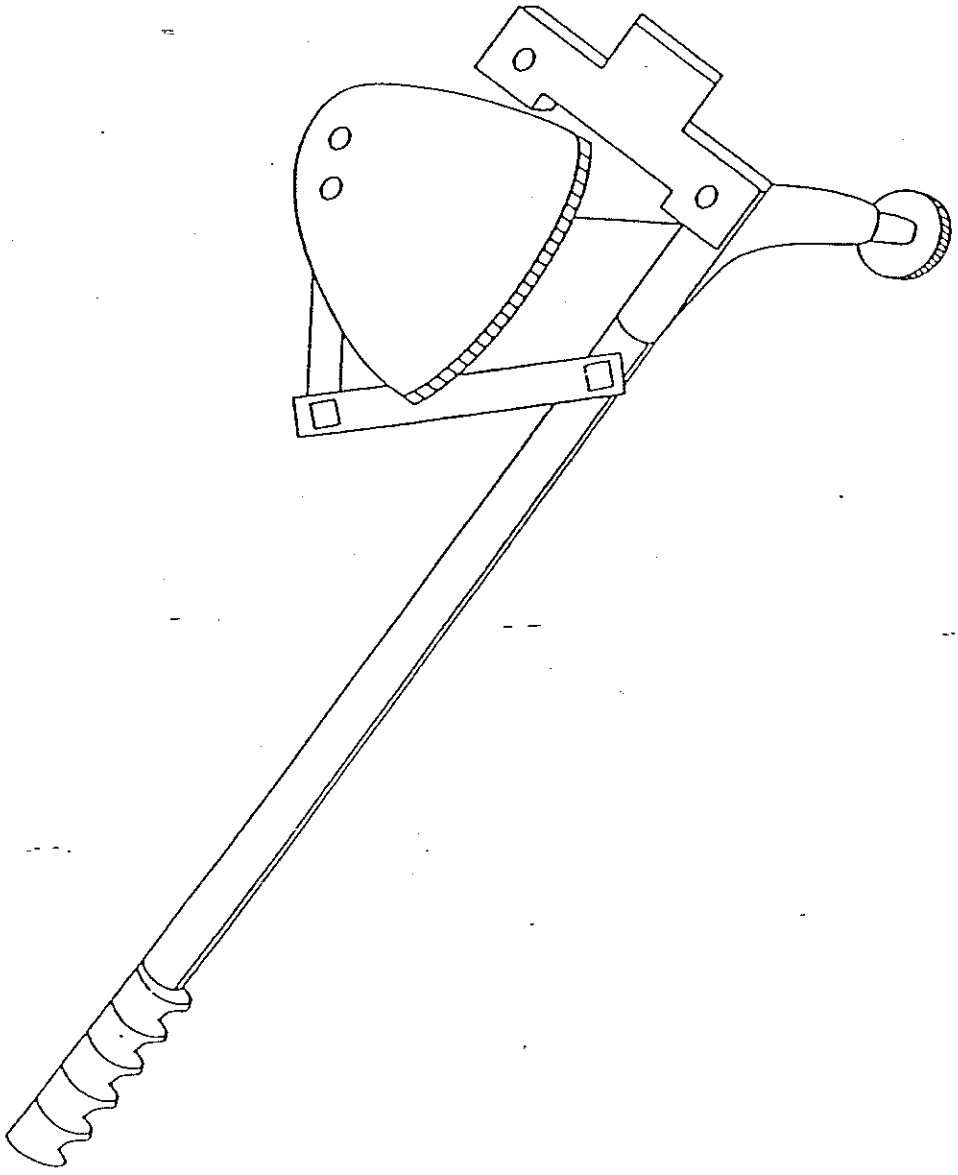


Figure 3: Hand Pick, Pickaxe, and Hand Spike

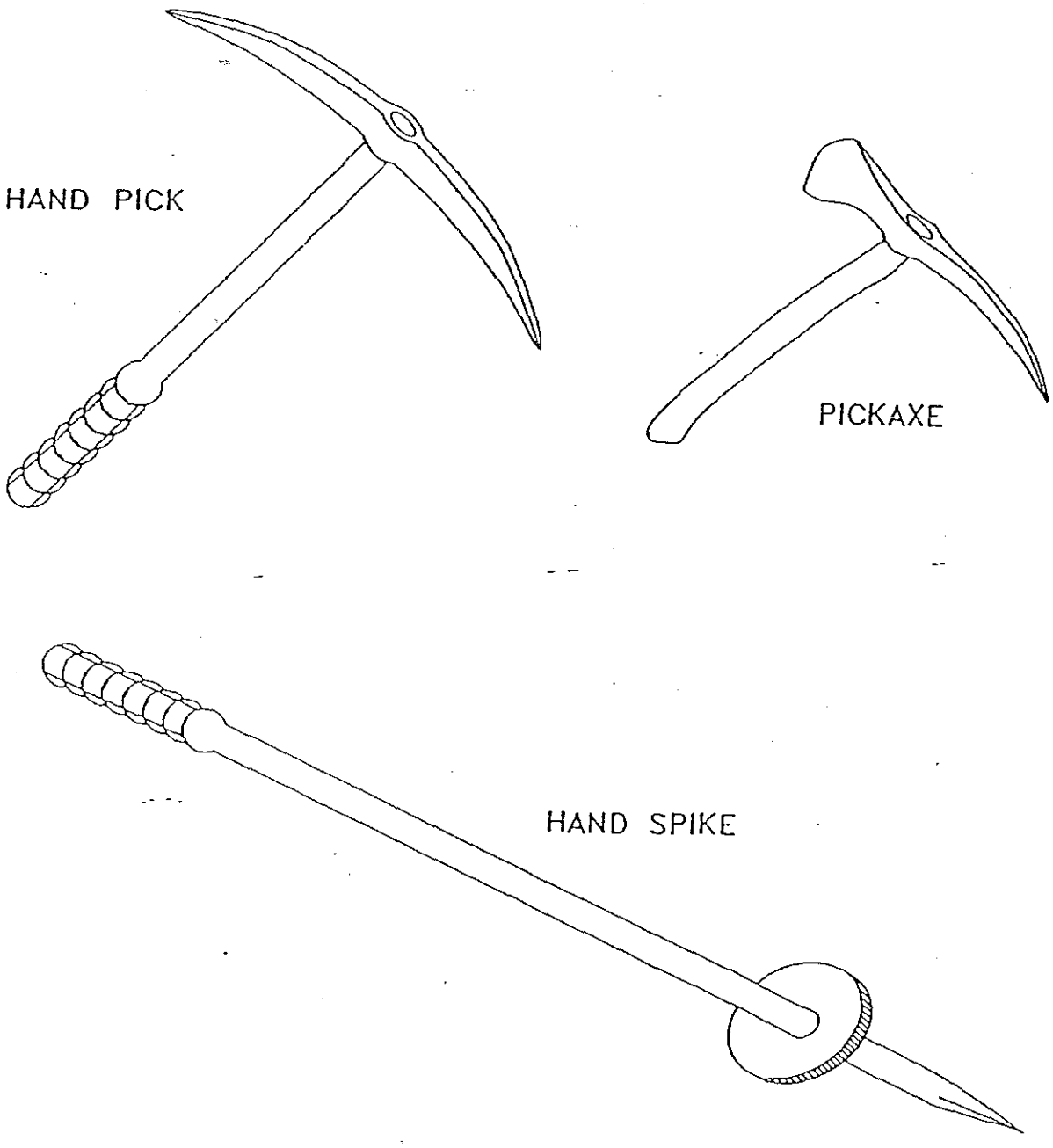


Figure 4: Backhoe Spike

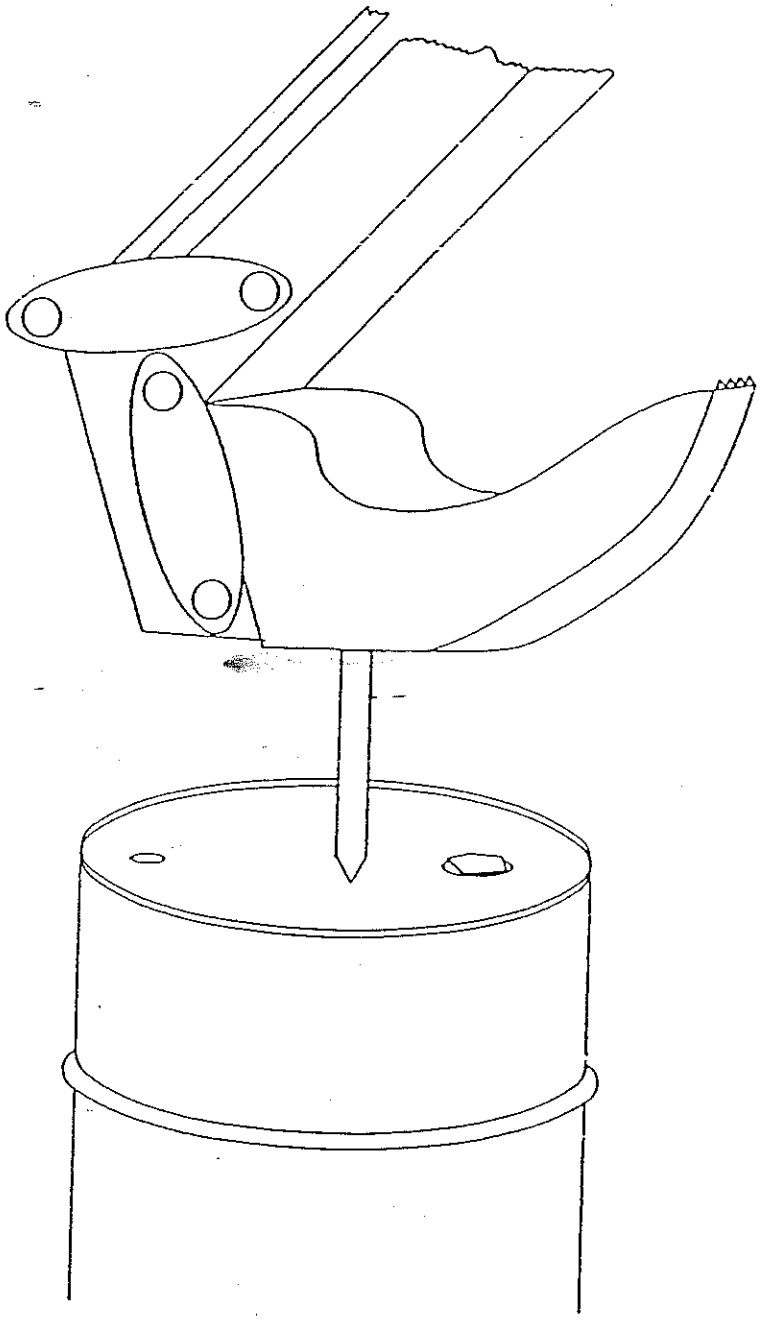


Figure 5: Hydraulic Drum Opener

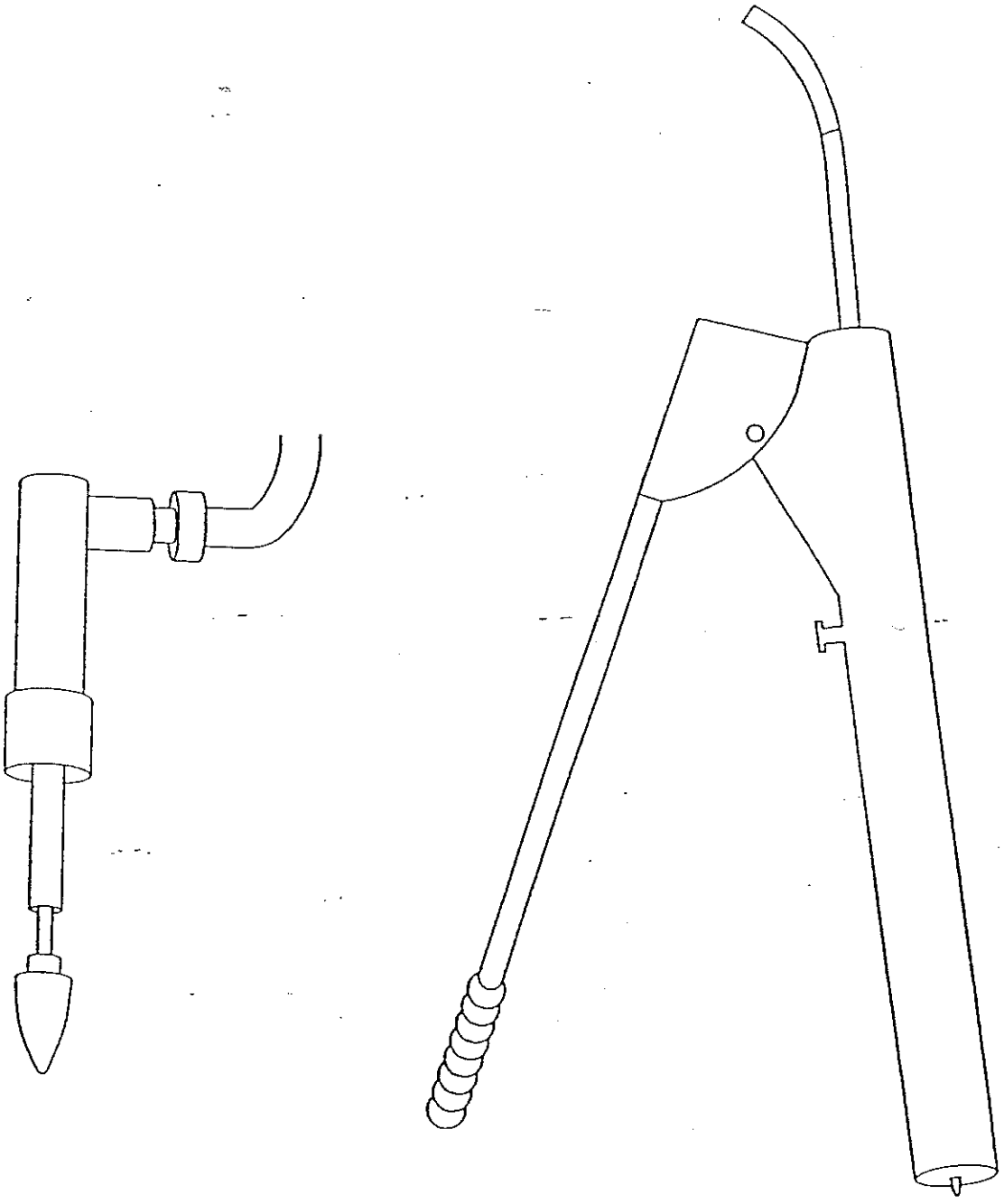


Figure 6: Pneumatic Bung Remover

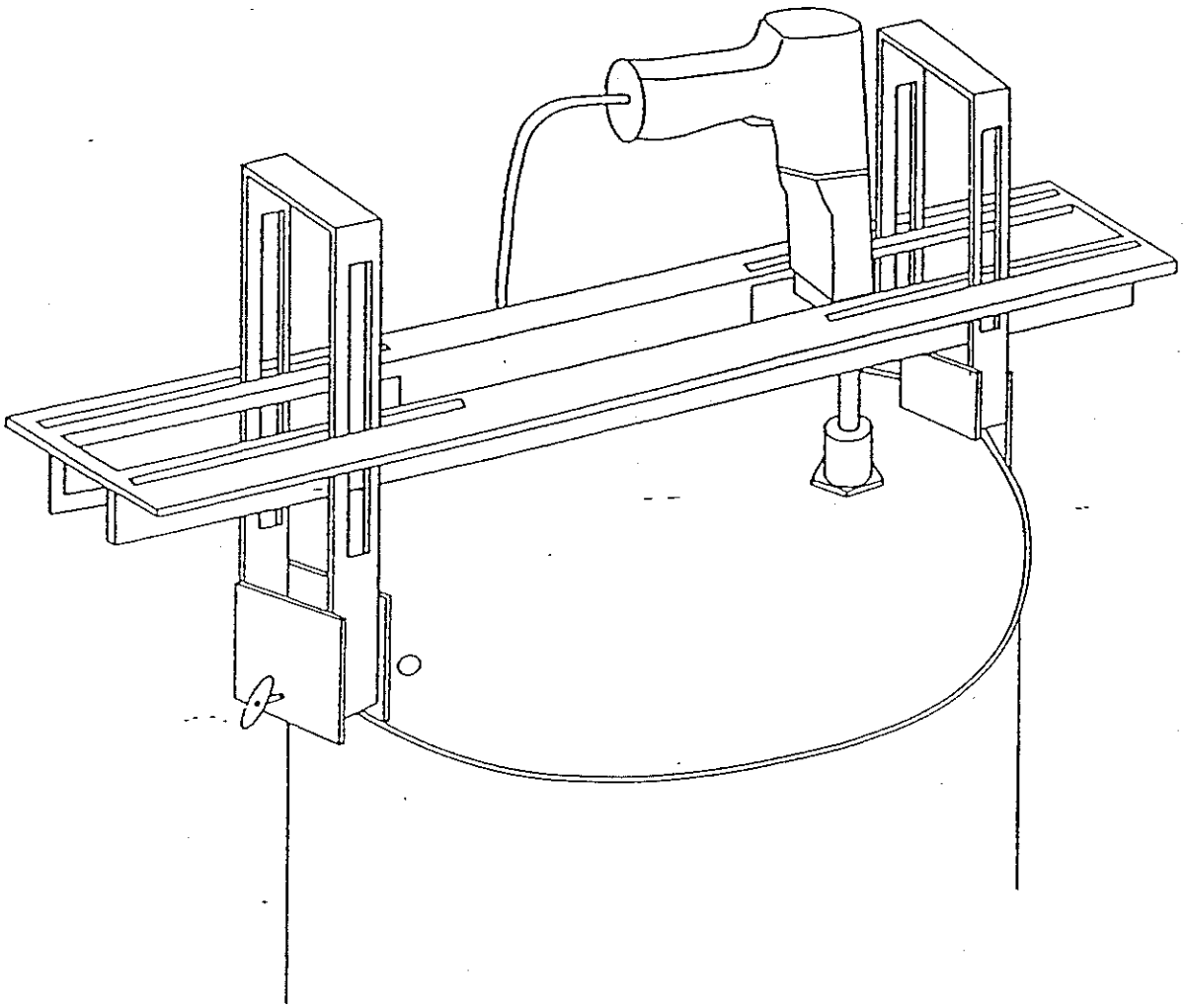
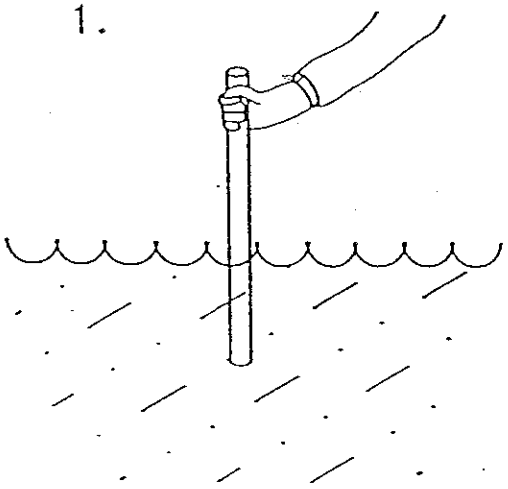


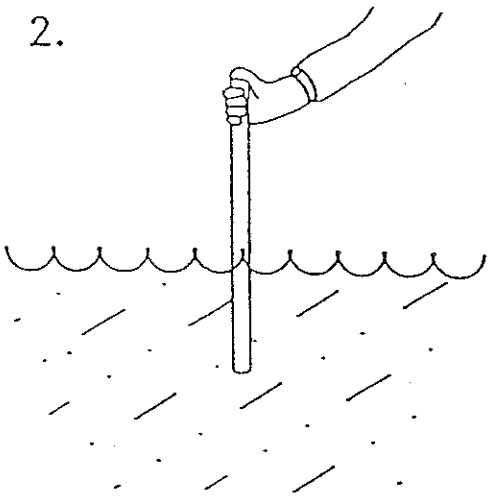
Figure 7: Glass Thief

1.



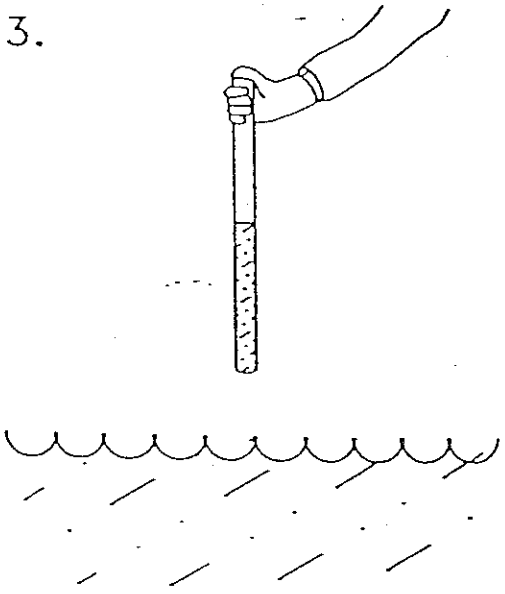
Insert open tube (thief) sampler in containerized liquid.

2.



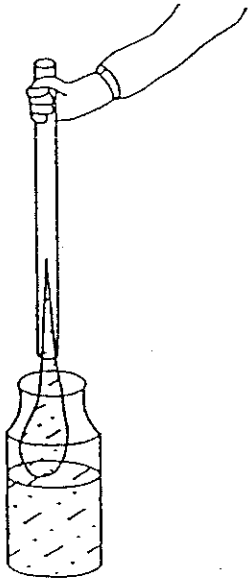
Cover top of sampler with gloved thumb.

3.



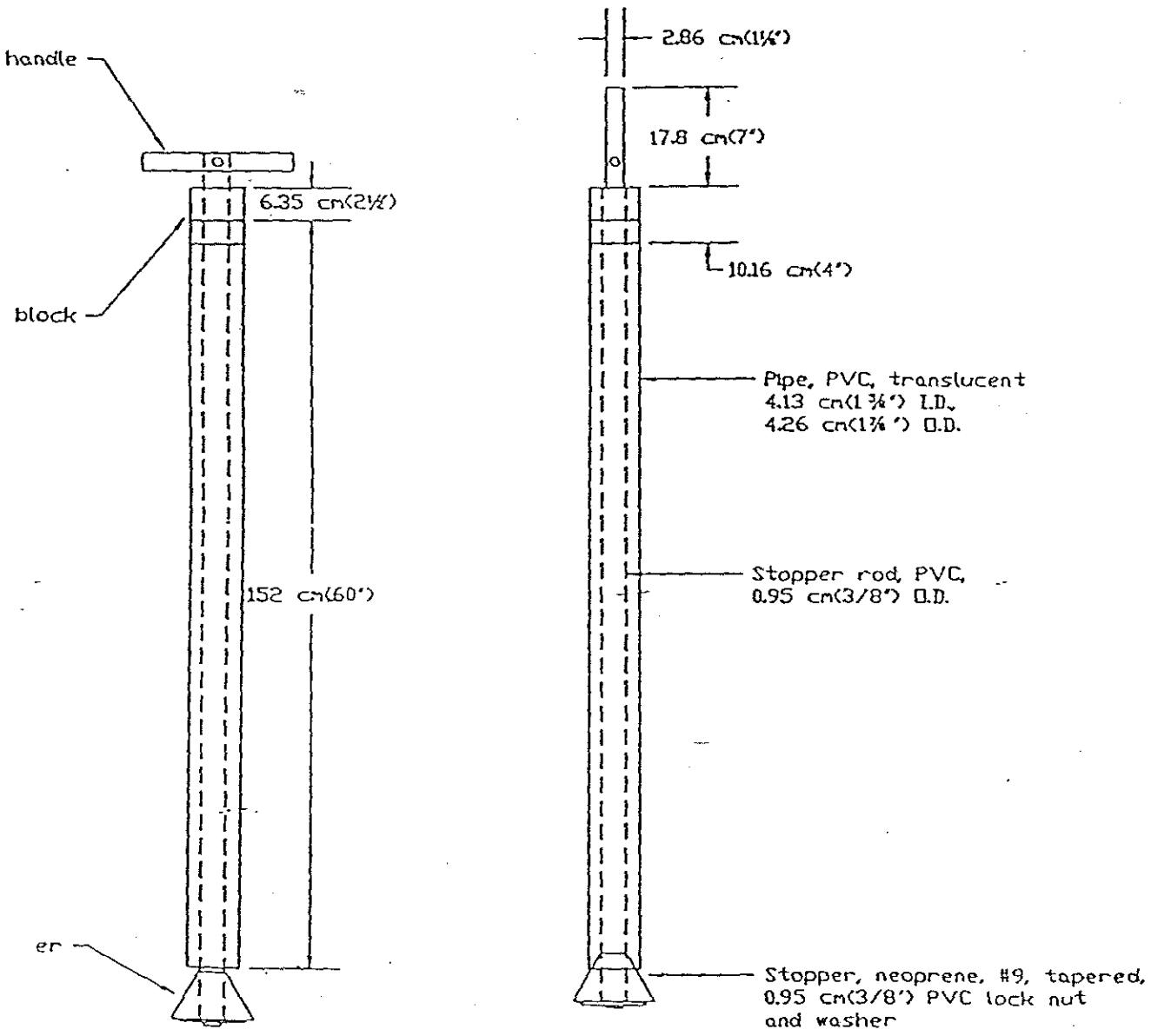
Remove open tube (thief) sampler from containerized liquid.

4.



Place open tube sampler over appropriate sample bottle and remove gloved thumb.

Figure 8: COLIWASA




SAMPLING POSITION CLOSED POSITION

# F201
# *ON-SITE WATER QUALITY TESTING*

# ON-SITE WATER QUALITY TESTING (FIELD PARAMETERS)

## 1.0 PURPOSE

This SOP describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific Conductance/Salinity
- Temperature
- Dissolved Oxygen Concentration (DO)
- Turbidity (Secchi Disc)

The first three are the usual field parameters; dissolved oxygen may be used in particular applications according to project requirements. In addition, the Secchi Disc is only used in slow-moving or stagnant waters, such as ponds.

## 2.0 SCOPE

These procedures are applicable for use in an on-site water quality monitoring program to be conducted during a Remedial Investigation or Site Investigation at a hazardous or nonhazardous site. The procedures and equipment described are applicable to nearly all aqueous samples, including potable well water, monitoring well water, surface water, leachate and drummed water, etc.

This procedure provides generic information for measuring the parameters listed above with instruments and techniques in common use. Since instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use.

## 3.0 DEFINITIONS

Conductance - A measurement of water's capacity for conveying electrical current and is directly related to the concentrations of ionized substances in the water. The units of measurement for conductance (mhos) are the inverse of ohms, the unit commonly used to express resistance. Conductivity and specific conductance are used synonymously.

Electrolytic Cell - An electrochemical cell in which electrical energy is supplied from an external source. This cell functions in much the same way as a galvanic cell, only in the opposite direction due to the extemal source of applied voltage.

Galvanic Cell - An electrochemical cell in which chemical energy is spontaneously converted to electrical energy. The electrical energy produced is supplied to an external circuit.

Oxidation - The process in which an atom or group of atoms loses electrons to achieve an increasing positive charge.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration. The range of pH is 0 to 14 standard units.

Resistance - A measure of the solution's ability to oppose the passage of electrical current. For metals and solutions, resistance is defined by Ohm's Law, $E = IR$, where E is the potential difference (in units of volts), I is the current (in units of Amperes), and R is the resistance (in units of ohms).

Secchi disc - A metal disc having four quadrants, two opposing ones painted black and the other two either white or unpainted. The Secchi disc is used to measure turbidity based on the depth of light penetration.

Turbidity - An optical property of water that causes light to be scattered or absorbed in the water, resulting in decrease in water transparency. It is a function of at least three variables: 1) dissolved chemicals, such as tannins, acids, or salts; 2) suspended particles, such as silt, clay, and organic matter; and, 3) density of microbial and planktonic life.

Salinity refers to the total amount of soluble salts in water, either naturally or added to the environment as pollutants.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other, approved procedures are developed. The Project Manager is responsible for determining which on-site water quality measurements shall be made, the data quality objectives (DQOs) for these measurements, and for ensuring that these measurements are made in accordance with project-specific plans.

Field Team Leader - The Field Team Leader is responsible for determining that these water quality measurement procedures are implemented in the field in accordance with this SOP, or in accordance with project-specific plans, and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures.

Sampling Personnel - It is the responsibility of the field sampling personnel to follow these procedures for collecting on-site water quality measurements including instrument calibration, quality control and recording of results, as well as care and maintenance of the instruments in the field.

## 5.0 PROCEDURES

The following sections provide general procedures for collecting pH, specific conductance/salinity, temperature, dissolved oxygen concentration and turbidity measurements.

## 5.1 Measurement of pH

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment, such as acid-base neutralization, water softening, and corrosion control, is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken.

Measurements of pH also can be used to check the quality and corrosivity of soil and solid waste samples. However, these samples must be immersed in water prior to analysis; specific measurement techniques for solids are not described here.

Two methods are given for pH measurement: the pH meter and pH indicator paper. The indicator paper is used when only a rough estimate of the pH is required; the pH meter is used when a more accurate measurement is needed. The response of a pH meter can be affected to a slight degree by high levels of colloidal or suspended solids, but the effect is usually small and generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In most cases, use of a pH meter will be required.

### 5.1.1 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution with the indicator compound on the paper. Depending on the indicator and the pH range of interest, a variety of different colors can be used. Typical indicators are weak acids or bases, or both. Process chemistry and molecular transformations leading to the color change are variable and complex.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on the establishment of a potential difference across a glass or other type of membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to hydrogen ion concentration can be generated and measured.

### 5.1.2 Equipment

The following equipment and reagents are needed for taking pH measurements:

- Portable pH meter, or pH indicator paper, such as Mydrion or Alkacid, to cover the pH range 2 through 12.
- Laboratory-prepared buffer solutions of pH 4, 7 and 10, or other buffers which bracket the expected pH range.

### 5.1.3 Measurement Techniques for Field Determination of pH

A. pH Meter

Standardization, calibration, and operation and maintenance shall be performed according to the manufacturers instructions. The following procedure is used for measuring pH with a pH meter:

1. The batteries shall be checked to make sure that they are fully charged and the instrument shall be calibrated prior to initiation of the field effort.

2. Immerse the tip of the electrodes in water overnight. If this is not possible due to field conditions, immerse the electrode tip in water for at least an hour before use. The electrode tip may be immersed in a rubber or plastic sack containing buffer solution for field transport or storage. This is not applicable for all electrodes as some must be stored dry.

3. Turn meter on and allow it to stabilize for 3 to 5 minutes.

4. The accuracy of the buffer solutions used for field and laboratory calibration shall be checked. Buffer solutions need to be changed often due to degradation upon exposure to the atmosphere. Select two pH buffers; 7, 4, and/or 10; in expected sample range and check temperatures of each. Record pertinent information in Field Logbook.

5. Make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).

6. Immerse the electrode(s) in a pH 7 buffer solution.

7. Adjust the temperature compensator to the proper temperature (on models with automatic temperature adjustment, immerse the temperature probe into the buffer solution). It is best to maintain the buffer solution at or near expected sample temperature before calibration, if possible.

8. Adjust the pH meter to read 7.0.

9. Remove the electrode(s) from the buffer and rinse well with distilled-deionized water. Immerse the electrode(s) in pH 4 or 10 buffer solution (depending on the expected pH of the sample) and adjust the slope control to read the appropriate pH. For best results, the standardization and slope adjustments shall be repeated at least once.

10. The calibration procedure should be performed:
    - Following significant ambient temperature changes
    - When meter reads erratically
    - At beginning and middle of each day of use

11. When the meter is moved to a new sampling location, a single-point calibration should be performed with pH 7 buffer.

12. Immerse the electrode(s) in the unknown solution, slowly stirring the probe until the pH stabilizes. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a chemical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. This must be clearly noted in the logbook.

13. After adjusting the temperature compensator to the sample temperature, read and record the pH of the solution. The pH value shall be recorded to the nearest 0.1 pH unit. Also record the sample temperature. All measurements shall be recorded in the Field Logbook.

14. Upon completion of measurement and removal of the electrode from the sample, the electrode shall be thoroughly rinsed with deionized water.

15. The electrode(s) shall remain immersed in deionized water when not in use.

The sample used for pH measurement shall never be saved for subsequent conductivity or chemical analysis. All pH electrodes leak small quantities of electrolytes (e.g., sodium or potassium chloride) into the solution. Precipitation of saturated electrolyte solution within the electrode, especially at colder temperatures, or in cold water, may result in slow electrode response. Any visual observation of conditions which may interfere with pH measurement, such as oily materials, or turbidity, shall be noted in the Field Logbook.

B. pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, or 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown, the investigation shall start with wide-range paper.

## 5.2 Measurement of Specific Conductance/Salinity

Conductance provides a measure of dissolved ionic species in water and can be used to suggest the direction and extent of migration of contaminants in groundwater or surface water. Salinity refers to the total amount of soluble salts in water, either naturally or added to the environment as pollutants. One basic measure of salinity is the ability of water to conduct electric current, and, therefore, a measurement of specific conductance provides a measurement of salinity and the same instrument can be used. Salinity measurements are important in ecological field investigations because flora and fauna can be limited in their distribution based on the salinity of the sampled waters.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of the ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance and salinity measurement soon after taking a sample, since temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect the specific conductance.

### 5.2.1 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions (cations) migrate toward the negative electrode (cathode), while the negatively charged ions (anions) migrate toward the positive electrode (anode). Most inorganic acids, bases and salts (such as hydrochloric acid, sodium carbonate, or sodium chloride, respectively) are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not disassociate in aqueous solution, conduct a current very poorly, if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell also may be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

### 5.2.2 Equipment

A portable conductivity meter, probe and thermometer are needed for taking specific conductance and salinity measurements. A variety of conductivity meters are available which also may be used to monitor salinity and temperatures. Probe types and cable lengths vary, so equipment may be obtained to meet the specific requirements of the sampling program.

### 5.2.3 Measurement Techniques for Specific Conductance/Salinity

Standardization, calibration, and operation and maintenance shall be performed according to manufacturers instructions. The steps involved in taking specific conductance and salinity measurements are listed below.

1. Check batteries to make sure they are fully charged and calibrate instrument before going into the field.

2. Calibrate the instrument daily when used. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used. Calibration information shall be recorded in the field logbook.

3. Turn meter on and allow it to stabilize for 3 to 5 minutes.

4. Pour approximately 50 to 100 ml of standard conductance solution (0.1 Molar Potassium Chloride), 1413 micromhos, into a rinsed plastic cup.

5. Determine temperature of reference solution.

6. Set temperature compensation knob on meter to temperature of reference solution. If the meter does not compensate for temperature variations, the corrections given in Attachment A shall be applied.

7. Rinse probe with distilled water and blot dry with paper towel.

8. Place probe in reference solution and adjust standard knob on meter to read value of reference solution. Confirm and document proper operation/reading against standard solution.

9. Remove probe and rinse with distilled water. Blot the end of the probe dry with paper towel.

10. This calibration procedure should be performed:
    - Following significant ambient temperature changes
    - When meter reads erratically
    - At beginning and middle of each day of use

11. Pour approximately 50 to 100 ml of sample into a rinsed plastic cup. Immerse the electrode in the sample and measure the conductivity and salinity. If specified, adjust the temperature setting to the sample temperature.

12. Read and record the results in the Field Logbook.

13. If the meter does not compensate for temperature variations, the corrections given in Attachment A shall be applied.

14. On some meters, specific conductivity and salinity measurements may need to be reported with the associated temperature measurement. If the conductivity and salinity has been corrected, the measurements shall be reported as "corrected to 25°C." (Attachment A)

    a. Do not take readings if the sample temperature is less than 10° C, because the calibration curve no longer follows a straight line below this temperature. If necessary, heat the sample in your vehicle to at least 10° C.

    b. Measure the sample temperature to the nearest 0.1° C to comply with SW-846.

    c. Only report results to the nearest two significant digits for the most circumstances, because of the inherent inaccuracy in the test and conversion procedure.

examples:

- a calculated reading of 2353 umhos/cm @ 25° C should be reported as 2400 umhos/cm @ 25° C
- a calculated reading of 2325 should be reported as 2300
- a calculated reading of 337 should be reported as 340
- etc.

## 5.3 Measurement of Temperature

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in-situ, or as quickly as possible in the field prior to sample collection. Collected water samples may rapidly equilibrate with the temperature of their surroundings.

### 5.3.1 Equipment

Temperature measurements may be taken with Thermistor, alcohol-toluene, mercury or bimetal thermometers. In addition, various meters such as specific conductance or dissolved oxygen meters, which have temperature measurement capabilities, may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature can be performed.

### 5.3.2 Measurement Techniques for Water Temperature

If a thermometer is used on a collected water sample:

1. Visually inspect thermometer to ensure that there is not a break in the mercury column. If there is a break, the spare thermometer will be visually inspected. If both thermometers have a break in the mercury, neither will be used until the break is corrected. This will be done by cooling the bulb until the mercury is contained within the bulb.

2. Immerse the thermometer in the sample until temperature equilibrium is obtained (1-3 minutes). To avoid the possibility of contamination, the thermometer shall not be inserted into samples which will undergo subsequent chemical analysis.

3. Record values in a Field Logbook to the nearest 0.5 or 0.1°C, depending on the measurement device used.

If a temperature meter or probe is to be used, the instrument shall be calibrated according to the manufacturer's recommendations with an approved thermometer.

## 5.4 Measurement of Dissolved Oxygen Concentration

Dissolved oxygen (DO) levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. Conversely, the growth of many aquatic organisms, as well as the rate of corrosivity, are dependent on the dissolved oxygen concentration. Thus, analysis for dissolved oxygen is a key test in water pollution and waste treatment process control. If at all possible, DO

measurements shall be taken in-situ, since concentration may show a large change in a short time, if the sample is not adequately preserved.

The method discussed here is limited to the use of dissolved oxygen meters only. Chemical methods of analysis (i.e., Winkler methods) are available, but require more equipment and greater sample manipulation. Furthermore, DO meters, using a membrane electrode, are suitable for highly polluted waters, because the probe is completely submersible. DO meters also are free from interference caused by color, turbidity, colloidal material or suspended matter.

### 5.4.1 Principles of Equipment Operation

DO probes normally are electrochemical cells that have two solid metal electrodes of different potential immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of higher nobility (the cathode) is positioned at the membrane. When a suitable potential exists between the two metals, reduction of oxygen to hydroxide ion (OH) occurs at the cathode surface. An electrical current is developed directly proportional to the rate of arrival of oxygen molecules at the cathode.

Since the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, but to leave the surface of the solution undisturbed.

DO probes are relatively free of interferences. Interferences that can occur are reactions with oxidizing gases (such as chlorine) or with gases such as hydrogen sulfide which are not easily depolarized from the indicating electrode. If gaseous interference is suspected, it shall be noted in the Field Logbook and checked if possible. Temperature, pressure, and salinity variations also can cause interference. Automatic temperature compensation normally is provided by the manufacturer. Attachment B presents variations of DO in water as a fraction of temperature and pressure. Salinity should be compensated in accordance with the manufactures instructions.

### 5.4.2 Equipment

The following, similar or equivalent, equipment is needed to measure dissolved oxygen concentration:

- YSI Model 57 dissolved oxygen monitor (or equivalent).
- Dissolved oxygen/temperature probe.
- Sufficient cable to allow the probe to contact the sample.

### 5.4.3 Measurement Techniques for Dissolved Oxygen Determination

Probes differ as to specifics of use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure the DO concentration.

1. Calibrate equipment and check batteries in the laboratory before going to the field.

2. The probe shall be conditioned in a water sample for as long as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.

3. The instrument shall be calibrated in the field before each measurement or group of closely spaced measurements by placing the probe in a water sample of known dissolved oxygen concentration (i.e., determined by Winkler method) or in a freshly air-saturated water sample of known temperature.

4. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane, either by stirring the sample, or placing the probe in a flowing stream. Probes without stirrers which are placed in wells should be moved up and down.

5. Record the dissolved oxygen content and temperature of the sample in a Field Logbook.

6. Recalibrate the probe when the membrane is replaced, or following similar maintenance, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable, since sample handling is not involved. This, however, may not always be practical. Be sure to record whether the liquid was analyzed in situ, or whether a sample was taken.

Special care shall be taken during sample collection to avoid turbulence which can lead to increased oxygen dissolution and positive test interferences.

## 5.5 Measurement of Turbidity Using a Secchi Disc

In combination with other parameters, turbidity can be a useful indicator of the likelihood of biological action in a water body. It can be used to determine the depth of light penetration of surface water and the distribution and intensity of photosynthesis in the body of water. Turbidity measurements shall be taken in-situ with a Secchi disc. It is noted that the Secchi disc is most accurate in slow-moving or stagnant waters and may not be appropriate for all sampling areas.

### 5.5.1 Equipment

Turbidity measurements may be taken with a Secchi disc. In addition, turbidity may be measured using a colorimeter or a spectrophotometer. These are ex-situ measurements conducted in a laboratory environment.

### 5.5.2 Measurement Techniques for Turbidity

Observations must be made through a shaded area of water surface.

- Standard conditions for the use of the Secchi disc are: 1) clear sky; (2) sun directly overhead; 3) shaded, protected side of boat or under a sun shade; 4) minimal waves or ripples; and, 5) any departure from these conditions should be specifically stated on field sheets.

- Rope accurately graduated in meters with 0.1 meter graduations for the first meter and 0.5 meters thereafter.
- Observer's eye should be 1 meter above the surface of the water.
- Observations should be made during the middle of the day.
- Lower the disc into the water, noting the depth at which it disappears, then lift the disc and note the depth at which it reappears. The average of the two readings is considered to be the limit of visibility and is recorded in a Field Logbook to the nearest 0.1 meter (first meter) or 0.5 meter, depending on the depth of visibility.

## 6.0 QUALITY ASSURANCE RECORDS

Quality assurance records for on-site water quality management consists principally of observations and measurements recorded in the field logbook.

## 7.0 REFERENCES

American Public Health Association, 1980. Standard Methods for the Examination of Water and Wastewater, 15th Edition, APHA, Washington, D.C.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

U.S. Geological Survey, 1984. National Handbook of Recommended Methods for Water Data Acquisition, Chapter 5: Chemical and Physical Quality of Water and Sediment. U.S. Department of the Interior, Reston, Virginia.

# ATTACHMENT A

## SPECIFIC CONDUCTANCE CONVERSION TABLE

ATTACHMENT A
SPECIFIC CONDUCTANCE CONVERSION TABLE

| Temperature Degrees C | Calculated Multiplier | Temperature Degrees C | Calculated Multiplier | Temperature Degrees C | Calculated Multiplier | Temperature Degrees C | Calculated Multiplier | Temperature Degrees C | Calculated Multiplier |
|---|---|---|---|---|---|---|---|---|---|
| 10 | 1.402 | 12 | 1.330 | 14 | 1.266 | 16 | 1.208 | 20 | 1.106 |
| 10.1 | 1.398 | 12.1 | 1.327 | 14.1 | 1.263 | 16.1 | 1.205 | 20.1 | 1.103 |
| 10.2 | 1.394 | 12.2 | 1.324 | 14.2 | 1.260 | 16.2 | 1.202 | 20.2 | 1.101 |
| 10.3 | 1.390 | 12.3 | 1.320 | 14.3 | 1.257 | 16.3 | 1.199 | 20.3 | 1.099 |
| 10.4 | 1.387 | 12.4 | 1.317 | 14.4 | 1.254 | 16.4 | 1.197 | 20.4 | 1.096 |
| 10.5 | 1.383 | 12.5 | 1.314 | 14.5 | 1.251 | 16.5 | 1.194 | 20.5 | 1.094 |
| 10.6 | 1.379 | 12.6 | 1.310 | 14.6 | 1.248 | 16.6 | 1.191 | 20.6 | 1.092 |
| 10.7 | 1.376 | 12.7 | 1.307 | 14.7 | 1.245 | 16.7 | 1.188 | 20.7 | 1.089 |
| 10.8 | 1.372 | 12.8 | 1.304 | 14.8 | 1.242 | 16.8 | 1.186 | 20.8 | 1.087 |
| 10.9 | 1.369 | 12.9 | 1.301 | 14.9 | 1.239 | 16.9 | 1.183 | 20.9 | 1.085 |
| 11 | 1.365 | 13 | 1.297 | 15 | 1.236 | 17 | 1.180 | 21 | 1.083 |
| 11.1 | 1.361 | 13.1 | 1.294 | 15.1 | 1.233 | 17.1 | 1.178 | 21.1 | 1.080 |
| 11.2 | 1.358 | 13.2 | 1.291 | 15.2 | 1.230 | 17.2 | 1.175 | 21.2 | 1.078 |
| 11.3 | 1.354 | 13.3 | 1.288 | 15.3 | 1.227 | 17.3 | 1.172 | 21.3 | 1.076 |
| 11.4 | 1.351 | 13.4 | 1.285 | 15.4 | 1.225 | 17.4 | 1.170 | 21.4 | 1.074 |
| 11.5 | 1.347 | 13.5 | 1.281 | 15.5 | 1.222 | 17.5 | 1.167 | 21.5 | 1.072 |
| 11.6 | 1.344 | 13.6 | 1.278 | 15.6 | 1.219 | 17.6 | 1.165 | 21.6 | 1.069 |
| 11.7 | 1.341 | 13.7 | 1.275 | 15.7 | 1.216 | 17.7 | 1.162 | 21.7 | 1.067 |
| 11.8 | 1.337 | 13.8 | 1.272 | 15.8 | 1.213 | 17.8 | 1.159 | 21.8 | 1.065 |
| 11.9 | 1.334 | 13.9 | 1.269 | 15.9 | 1.210 | 17.9 | 1.157 | 21.9 | 1.063 |
| 22 | 1.061 | 24 | 1.019 | 26 | 0.981 | 28 | 0.946 | 30 | 0.913 |
| 22.1 | 1.059 | 24.1 | 1.017 | 26.1 | 0.979 | 28.1 | 0.944 | 30.1 | 0.911 |
| 22.2 | 1.057 | 24.2 | 1.016 | 26.2 | 0.978 | 28.2 | 0.942 | 30.2 | 0.910 |
| 22.3 | 1.054 | 24.3 | 1.014 | 26.3 | 0.976 | 28.3 | 0.941 | 30.3 | 0.908 |
| 22.4 | 1.052 | 24.4 | 1.012 | 26.4 | 0.974 | 28.4 | 0.939 | 30.4 | 0.907 |
| 22.5 | 1.050 | 24.5 | 1.010 | 26.5 | 0.972 | 28.5 | 0.937 | 30.5 | 0.905 |
| 22.6 | 1.048 | 24.6 | 1.008 | 26.6 | 0.970 | 28.6 | 0.936 | 30.6 | 0.903 |
| 22.7 | 1.046 | 24.7 | 1.006 | 26.7 | 0.969 | 28.7 | 0.934 | 30.7 | 0.902 |
| 22.8 | 1.044 | 24.8 | 1.004 | 26.8 | 0.967 | 28.8 | 0.932 | 30.8 | 0.900 |
| 22.9 | 1.042 | 24.9 | 1.002 | 26.9 | 0.965 | 28.9 | 0.931 | 30.9 | 0.899 |

ATTACHMENT A (Continued)
SPECIFIC CONDUCTANCE CONVERSION TABLE

| Temperature Degrees C | Calculated Multiplier | Temperature Degrees C | Calculated Multiplier | Temperature Degrees C | Calculated Multiplier | Temperature Degrees C | Calculated Multiplier | Temperature Degrees C | Calculated Multiplier |
|---|---|---|---|---|---|---|---|---|---|
| 23 | 1.040 | 25 | 1.000 | 27 | 0.963 | 29 | 0.929 | 31 | 0.897 |
| 23.1 | 1.038 | 25.1 | 0.998 | 27.1 | 0.961 | 29.1 | 0.927 | 31.1 | 0.896 |
| 23.2 | 1.036 | 25.2 | 0.996 | 27.2 | 0.960 | 29.2 | 0.926 | 31.2 | 0.894 |
| 23.3 | 1.034 | 25.3 | 0.994 | 27.3 | 0.958 | 29.3 | 0.924 | 31.3 | 0.893 |
| 23.4 | 1.032 | 25.4 | 0.992 | 27.4 | 0.956 | 29.4 | 0.922 | 31.4 | 0.891 |
| 23.5 | 1.029 | 25.5 | 0.991 | 27.5 | 0.954 | 29.5 | 0.921 | 31.5 | 0.890 |
| 23.6 | 1.027 | 25.6 | 0.989 | 27.6 | 0.953 | 29.6 | 0.919 | 31.6 | 0.888 |
| 23.7 | 1.025 | 25.7 | 0.987 | 27.7 | 0.951 | 29.7 | 0.918 | 31.7 | 0.887 |
| 23.8 | 1.023 | 25.8 | 0.985 | 27.8 | 0.949 | 29.8 | 0.916 | 31.8 | 0.885 |
| 23.9 | 1.021 | 25.9 | 0.983 | 27.9 | 0.948 | 29.9 | 0.914 | 31.9 | 0.884 |

Notes:

- Do not make specific conductance measurements at temperatures below 10° C.
- Measure temperature to the nearest 0.1° C.
- Report all conductivities at 25° C, to two significant digits.
- This conversion table is based on a temperature coefficient of 0.0191 (as per SW-846) and a cell constant of 1, where the ratio of conductivity at 25 C to the conductivity at temperature t° C equals 1/(1+0.0191[t-25]).
- The temperature coefficient and cell constants are only approximate, actual values may differ.
- The more the temperature deviates from 25° C, the greater the uncertainty in applying the temperature correction.

# ATTACHMENT B

# VARIATION OF DISSOLVED OXYGEN CONCENTRATION IN WATER AS A FUNCTION OF TEMPERATURE AND SALINITY

| Temperature °C | 2.Dissolved Oxygen mg/l<br>2.1.Chloride Concentration in Water<br>2.1.1.0<br>2.1.2.5,000<br>2.1.3.10,000<br>2.1.4.15,000<br>2.1.5.20,000<br>2.2.Difference/<br>100 mg chloride | | | | | |
|---|---|---|---|---|---|---|
| 0 | 14.6 | 13.8 | 13.0 | 12.1 | 11.3 | 0.017 |
| 1 | 14.2 | 13.4 | 12.6 | 11.8 | 11.0 | 0.106 |
| 2 | 13.8 | 13.1 | 12.3 | 11.5 | 10.8 | 0.015 |
| 3 | 13.5 | 12.7 | 12.0 | 11.2 | 10.5 | 0.015 |
| 4 | 13.1 | 12.4 | 11.7 | 11.0 | 10.3 | 0.014 |
| 5 | 12.8 | 12.1 | 11.4 | 10.7 | 10.0 | 0.014 |
| 6 | 12.5 | 11.8 | 11.1 | 10.5 | 9.8 | 0.014 |
| 7 | 12.2 | 11.5 | 10.9 | 10.2 | 9.6 | 0.013 |
| 8 | 11.9 | 11.2 | 10.6 | 10.0 | 9.4 | 0.013 |
| 9 | 11.6 | 11.0 | 10.4 | 9.8 | 9.2 | 0.012 |
| 10 | 11.3 | 10.7 | 10.1 | 9.6 | 9.0 | 0.012 |
| 11 | 11.1 | 10.5 | 9.9 | 9.4 | 8.8 | 0.011 |
| 12 | 10.8 | 10.3 | 9.7 | 9.2 | 8.6 | 0.011 |
| 13 | 10.6 | 10.1 | 9.5 | 9.0 | 8.5 | 0.011 |
| 14 | 10.4 | 9.9 | 9.3 | 8.8 | 8.3 | 0.010 |
| 15 | 10.2 | 9.7 | 9.1 | 8.6 | 8.1 | 0.010 |

| Temperature °C | 2.Dissolved Oxygen mg/l<br>2.1.Chloride Concentration in Water<br>2.1.1.0<br>2.1.2.5,000<br>2.1.3.10,000<br>2.1.4.15,000<br>2.1.5.20,000<br>2.2.Difference/<br>100 mg chloride | | | | | |
|---|---|---|---|---|---|---|
| 16 | 10.0 | 9.5 | 9.0 | 8.5 | 8.0 | 0.010 |
| 17 | 9.7 | 9.3 | 8.8 | 8.3 | 7.8 | 0.010 |
| 18 | 9.5 | 9.1 | 8.6 | 8.2 | 7.7 | 0.009 |
| 19 | 9.4 | 8.9 | 8.5 | 8.0 | 7.6 | 0.009 |
| 20 | 9.2 | 8.7 | 8.3 | 7.9 | 7.4 | 0.009 |
| 21 | 9.0 | 8.6 | 8.1 | 7.7 | 7.3 | 0.009 |
| 22 | 8.8 | 8.4 | 8.0 | 7.6 | 7.1 | 0.008 |
| 23 | 8.7 | 8.3 | 7.9 | 7.4 | 7.0 | 0.008 |
| 24 | 8.5 | 8.1 | 7.7 | 7.3 | 6.9 | 0.008 |
| 25 | 8.4 | 8.0 | 7.6 | 7.2 | 6.7 | 0.008 |
| 26 | 8.2 | 7.8 | 7.4 | 7.0 | 6.6 | 0.008 |
| 27 | 8.1 | 7.7 | 7.3 | 6.9 | 6.5 | 0.008 |
| 28 | 7.9 | 7.5 | 7.1 | 6.8 | 6.4 | 0.008 |
| 29 | 7.8 | 7.4 | 7.0 | 6.6 | 6.3 | 0.008 |
| 30 | 7.6 | 7.3 | 6.9 | 6.5 | 6.1 | 0.008 |

# F202
# *WATER LEVEL, WATER-PRODUCT LEVEL, AND WELL DEPTH MEASUREMENTS*

# WATER LEVEL, WATER-PRODUCT LEVEL MEASUREMENTS, AND WELL DEPTH MEASUREMENTS

## 1.0 PURPOSE 1.0 PURPOSE

The purpose of this procedure is to describe the method of determining various down-hole measurements: groundwater levels and product (or non-aqueous phase liquid, NAPL) levels, if present, and total depth of groundwater monitoring wells and piezometers.

## 2.0 SCOPE

The methods described in this SOP generally are applicable to the measurement of groundwater levels, product or NAPL levels, and well depths in monitoring wells and piezometers.

## 3.0 DEFINITIONS

None.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed.

Field Team Leader - The Field Team Leader is responsible for ensuring that these procedures are implemented in the field, and for ensuring that personnel performing these activities have been briefed and trained to execute these procedures.

Sampling Personnel - It is the responsibility of the sampling personnel to follow these procedures or to follow documented, project-specific procedures as directed by the Field Team Leader and/or the Project Manager. The sampling personnel are responsible for the proper acquisition of down-hole measurements.

## 5.0 PROCEDURES

Calculations of groundwater elevations and product or NAPL interface level measurements collected from a monitoring well give an indication of:

- The horizontal hydraulic gradient and the direction of groundwater flow.
- The vertical hydraulic gradient, if well nests are used (i.e., the direction of groundwater flow in the vertical plane).
- Floating or sinking product thicknesses which are also known as Light Non-Aqueous Phase Liquids (LNAPLs) and Dense Non-Aqueous Phase Liquids (DNAPLs), respectively.

This information, when combined with other site specific information such as hydraulic conductivity or transmissivity, extent of contamination, and product density, may be used to estimate the rate of contaminant movement or source areas, etc.

Well depth is one of the factors used to determine the zone that a well monitors. Well depth also is used in the calculation of purge volumes as discussed in SOP F104, Groundwater Sample Acquisition.

The following sections briefly discuss the procedures for measuring groundwater levels, product or NAPL levels, and well depth. For all of the procedures discussed, it is assumed that the measurement will be taken from the top of the PVC or stainless steel casing (though other measuring points can be used), and that horizontal and vertical control is available for each well through a site survey, such that measurements may be converted to elevations above Mean Sea Level (MSL) or some other consistent datum. A permanent notch, placed on the inner PVC or stainless steel casing by the surveyor will facilitate consistent water level measurements.

The manufacturer's instructions for all equipment referenced herein should be read by the equipment operator(s) and accompany the equipment to the field.

## 5.1 Water Level Measurement

Water levels in groundwater monitoring wells shall be measured from the permanent point indicated at the top of the inner casing (the surveyed elevation point, as marked by the surveyor), unless otherwise specified in the project plans, using an electronic water level measuring device (water level indicator). The point of measurement will be documented in the Field Logbook if different from the top of the inner casing. The reason for deviating from the measurement point should also be noted.

Water levels are measured by lowering the probe into the well until the device indicates that water has been encountered, usually with either a constant buzz, or a light, or both. The water level is recorded to the nearest foot (0.01) using the graduated markings on the water level indicator cord. This measurement, when subtracted from the measuring point elevation, yields the groundwater elevation.

Groundwater levels shall always be measured to the nearest 0.01 foot. However, reporting of water level elevations depends on the accuracy of the vertical control (typically either 0.1 or 0.01 foot).

## 5.2 Product or NAPL Level Measurements

The procedure for product or NAPL level measurement is nearly identical to that for groundwater elevation measurements. The only differences are the use of an interface probe that detects both NAPLs and water, and the indication signal given by the measurement device. Typically, encountering NAPLs in a monitoring well is indicated by a constant sound. When water is encountered, the signal becomes an alternating on/off beeping sound. This allows for the collection of measurements for both the top of the NAPL layer in a well and the water/NAPL interface.

The apparent water table elevation below the product level will be determined by subtracting the "depth to water" from the measuring point elevation. The corrected water table elevation will then be calculated using the following equation:

$$WTE_c = WTE_a + (\text{Free Product Thickness} \times 0.80)$$

Where:

| | | |
|---|---|---|
| $WTE_c$ | = | Corrected water table elevation |
| $WTE_a$ | = | Apparent water table elevation |
| 0.80 | = | Average value for the density of petroleum hydrocarbons. Site-specific data will be used where available. |

## 5.3 Well Depth Measurements

Well depths typically are measured using a weighted measuring tape. A water level meter may also be used. The tape is lowered down the well until resistance is no longer felt, indicating that the weight has touched the bottom of the well. The weight should be moved in an up and down motion a few times so that obstructions, if present, may be bypassed. The slack in the tape then is collected until the tape is taut. The well depth measurement is read directly off of the measuring tape, at the top of the PVC or stainless steel casing, to the nearest 0.01-foot and recorded in the Field Logbook. If a water level indicator is used, add the distance from the bottom of the probe to the point where water levels are measured.

## 5.4 Decontamination of Measuring Devices

Water level indicators, interface probes and weighted measuring tapes that come in contact with groundwater must be decontaminated using the following steps after use in each well:

- Rinse with potable water
- Rinse with deionized water
- Rinse with Methanol or Isopropanol
- Rinse with deionized water

Portions of the water level indicators or other similar equipment that do not come into contact with groundwater, but may encounter incidental contact during use, need only undergo potable water and deionized water rinses.

## 6.0 QUALITY ASSURANCE RECORDS

The Field Logbook shall serve as the quality assurance record for water, product level or well depth measurements.

# F203
# *PHOTOIONIZATION DETECTOR (PID)*

# PHOTOIONIZATION DETECTOR (PID)
# HNu MODELS PI 101 and DL 101

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information for using the HNu Model PI 101 or DL 101 photoionization detector (PID), or an equivalent or similar instrument, in the field. Calibration and operation, along with field maintenance will be included in this SOP.

## 2.0 SCOPE

This procedure provides information on the field operation and general maintenance of the HNu PID. Application of the information contained herein will ensure that this type of field monitoring equipment will be used properly. Review of the manufacturer's instruction manual is necessary for more complete information.

These procedures refer only to monitoring for health and safety. The methods are not directly applicable to surveillance of air quality for analytical purposes.

## 3.0 DEFINITIONS

Ionization Potential - In this case, a numeric equivalent that expresses the amount of energy needed to replace an electron with a photon. This energy is further defined in terms of electron volts (eV).

PID - Photoionization Detector

ppm - parts per million: parts of vapor or gas per million parts of air (directly proportional to calibration gas).

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for selecting qualified individuals for the monitoring activities.

Project Health and Safety Officer (PHSO) - The Project Health and Safety Officer is responsible for developing a site-specific Health and Safety Plan (HASP) which specifies air monitoring requirements.

Field Team Leader - It is the responsibility of the Field Team Leader to implement these procedures in the field, and to ensure that the Field Investigation Personnel performing air monitoring activities, have been briefed and trained to execute these procedures before the start of site operations.

Site Health and Safety Officer (SHSO) - The SHSO is responsible for ensuring that the specified air monitoring equipment is on site, calibrated, and used correctly by the Field Personnel. The SHSO will coordinate these activities with the Field Team Leader.

Field Investigation Personnel - It is the responsibility of the Field Investigation Personnel to follow these procedures or to follow documented project-specific procedures as directed by the Field Team Leader/Site Health and Safety Officer. The Field Investigation Personnel are responsible for documenting all air monitoring results in both the Field Logbook and the daily Realtime Air Monitoring Log during each field investigation.

## 5.0 PROCEDURES

The HNu PID utilizes the principle of photoionization whereby contaminant molecules enter the ion chamber and electrons are displaced by ultraviolet photons producing positive ions. These displaced positive ions are in turn collected on a special electrode. As the positive ions collect on the electrode, they create an electrical current which is amplified and displayed on the meter as a concentration in parts per million (ppm).

The HNu PID is only effective for contaminants that have ionization potentials (IP) of less than or equal to the electron volt (eV) capacity of the lamp (i.e., methane, having an IP of 12.98 eV, will not be detected at a lamp potential of 11.7 eV). The standard lamp is 10.2 eV with optional lamps of 9.5 eV and 11.7 eV, respectively. For the PI 101 the span settings should be as follows: 1.0 for 9.5 eV lamps; 9.8 for 10.2 eV lamps; and 5.0 for 11.7 eV lamps. During calibration, these span settings will be adjusted as necessary, using the span control knob.

The following subsections will discuss HNu PID calibration, operation, and maintenance. These sections, however, should not be used as a substitute for the manufacturer's instruction manual.

### 5.1 Calibration

For calibration purposes, the following items will be needed:

- Gas cylinder containing 95 to 100 parts per million (ppm) of isobutylene, balance in air.
- A 0.30 liters per minute regulator.
- Connector tubing.
- Screwdriver set.
- Photoionization Detector (PID) Calibration Form.

Prior to each use, make sure that the battery is fully charged, the ultraviolet lamp is working, and that the fan is operating and drawing air into the probe (fan operates at approximately 100-200 cc/minute for the PI 101 and 225 cc/minute for the DL 101). Procedures for completing these preliminary activities are given in the manufacturer's instruction manual.

PI 101

To calibrate the PI 101, the steps provided below should be followed. For an itemized description of the calibration process, refer to Section 3-5 in the manufacturer's instruction manual. The PI 101 should be calibrated on a daily basis.

- Turn the function control switch to the standby position and zero the instrument by turning the zero adjustment knob to align the indicator needle with zero on the readout meter.

- Set the range on the PI 101 and allow the instrument to warm up a few minutes before calibrating. Choices for range are 0-20, 0-200, and 0-2,000 ppm, respectively. Range choice must take into account the concentration of the calibration gas. For example, if you are using a concentration of 100 ppm isobutylene as the calibration gas, your range should be set on the 0-200 scale. If you have to zero the instrument in the desired range, record background if present.

- Attach tubing to the regulator

- Attach the free end of the tubing to the probe and turn on the calibration gas.

- Calibrate the PI 101 to benzene equivalents. Using the 10.2 eV (lamp) probe and 100 ppm isobutylene, the meter should read 56 units. Using the 11.7 eV (lamp) probe and 100 ppm isobutylene, the meter should read 65 units. If the reading on the meter is not ±5 percent of the concentration of the calibration gas, adjust the span setting knob until the meter reads accordingly. If after adjusting the span setting knob the readout meter is still not responding, refer to the manufacturer's instruction manual. Also, when the PI 101 is calibrated it should respond to a minimum of 90 percent of the concentration of the calibration gas within three seconds after introduction of that gas. If proper calibration cannot be obtained, internal calibration may be required. Note, only qualified personnel should perform internal calibrations.

- Record the calibration on the "Photoionization Detector (PID) Calibration Form".

DL 101

To calibrate the DL 101, the steps provided below should be followed. For an itemized description of the calibration process, refer to Section 4.4 in the manufacturer's instruction manual.

- Press and release the POWER button on the keypad and wait for the screen to stabilize then press the CALIBRATE key until "Calibrate?" appears. At this point press the ENTER key until "Elec_Zero? Yes" appears on the screen in which case you will press the ENTER key, again, to confirm the electronic zero.

- The display will now read "CE/ENT/EXIT Conc = ____ ppm" which requires the concentration of the calibration gas (noted on the side of the calibration gas bottle) to be entered on the keypad. The display will prompt you to "Attach gas to probe and /ENTER/" so attach tubing to probe (use the calibration gas humidifier in high humidity environments), open valve, and press ENTER key. Press ENTER again when "Press ENTER when Ready: xxx ppm" appears on screen. This will cause "Calibrating...Please Wait" to appear on screen.

Note: This calibration is effective when the instrument is in the Survey Mode, which is the default mode. For calibrations other the one described, or if proper calibration cannot be obtained, refer to the manufacturer's instruction manual.

- For calibrations using an alternate gas or span values, refer to Section 4.5 of the manufacturer's instruction manual.

- Record the calibration on the "Photoionization Detector (PID) Calibration Form" which accompanies each DL 101.

## 5.2 Operation

### PI 101

Note: IMPORTANT - The PI 101 should be "zeroed" in a fresh air environment if at all possible. If there is a background concentration, it must be documented and then zeroed out.

- Prior to each use of the PI 101, check that the battery is fully charged by turning the dial to BATT and making sure that it is within range. Also make sure that the ultraviolet lamp and the fan are working properly.

- Select your desired range. PI 101 ranges consists of a 0-20, 0-200, and 0-2,000 ppm, respectively. Consult with the Field Team Leader for more information when choosing the appropriate range, however, in most instances the range will be set initially at 0-20.

- When PI 101 is used intermittently, turn knob to STANDBY to help in extending the life of the UV lamp when operating in a low humidity environment. Otherwise, leave the knob set to the range desired so that the UV lamp will "burn off" any accumulated moisture.

Note: When using the PI 101, make sure that the probe does not contact water or soil during sampling. This will cause erroneous readings and will possibly damage the instrument.

### DL 101

The DL 101 is designed to default to the survey mode when initially powered up, therefore once the calibration has been completed, the instrument is ready to go. Within the survey mode several options are available, briefly these options include:

1. The Site Function

The Site function assigns a number to a site that is being analyzed. Press the Site Key on the keypad to enter a specific site number, or press the gray button on the rear of the probe to increment a site number.

2. Logging Data

The Log function stores data in memory. To log data, press the Log key on the keypad or the Log button on the back of the probe. "Log" will appear in the upper right corner of the display when activated and disappears when not activated. To turn logging off, press either the Log key on the keypad or the red Log button on the rear of the probe.

- The DL 101 allows for the interchanging of different voltage lamps, however, refer to the manufacturer's instructions before attempting to change the lamp.

- The DL 101 also offers three other modes of operation, the Hazardous Waste Mode, the Industrial Hygiene Mode, and the Leak Detection Mode. Each of these modes increases the range of capabilities for this instrument which is covered in detail in the manufacturer's instruction manual.

Note: When using the DL 101, make sure that the probe does not contact water or soil during sampling. This will cause erroneous readings and will possibly damage the instrument.

## 5.3 Interferences and Potential Problems

A number of factors can affect the response of the PI 101 and DL 101.

- High humidity can cause lamp fogging and decreased sensitivity. This can be significant when soil moisture levels are high, or when monitoring a soil gas well that is accessible to groundwater.

- High concentrations of methane can cause a downscale deflection of the meter.

- High and low temperature, electrical fields, FM radio transmission, and naturally occurring compounds, such as terpines in wooded areas, will also affect instrument response.

## 5.4 Maintenance

The best way to keep an HNu PID operating properly is to keep it as clean as possible. HNu PID's should be decontaminated or wiped down daily or after each use, as appropriate.

### Corrective Maintenance

- The ultraviolet lamp should be periodically cleaned using a special compound supplied by HNu Systems, Inc. for the 10.2 eV lamp, and a chlorinated solvent such as 1,1,1-trichloroethane for the 11.7 eV lamp. Consult the manufacturer's instruction manual for specific cleaning instructions.

- The ionization chamber can be periodically cleaned with methyl alcohol and a swab.

Note: UV lamp and ion chamber cleaning is accomplished by following the procedures outlined in Section 5.2, however, this should only be performed by trained personnel.

- Documenting the HNu PID's observed symptoms and then referring to the manufacturer's instruction manual section on troubleshooting (Section 6.0) also can be employed. If this does not work, the Field Team Leader should be consulted for an appropriate course of action.

Repair and Warranty Repair - HNu PID's have different warranties for different parts, so documenting the problem and sending it into the manufacturer assists in expediting repair time and obtaining appropriate warranty service.

### 5.5 Shipping and Handling

Following is information regarding the transport of the HNu PID meter and calibration gas.

- If the HNu PID is to be carried on aircraft, the calibration gas must be removed from the carrying case as cylinders of compressed gas are not permitted on passenger aircraft. The calibration gas should either be shipped to the site of its intended use, or purchased locally.

- Shipping of the calibration gas requires the completion of a form (specified by the shipping company) that identifies the package as a compressed gas. Compressed gas stickers must be affixed to the package.

## 6.0 QUALITY ASSURANCE RECORDS

Quality assurance records will be maintained for each air monitoring event. The following information shall be recorded in the Field Logbook.

- Identification - Site name, location, activity monitored, (surface water sampling, soil sampling, etc.) serial number, time, resulting concentration, comments and identity of air monitoring personnel.

- Field observations - Appearance of sampled media (if definable).

- Additional remarks (e.g., the HNu PID meter had wide range fluctuations during air monitoring activities).

## 7.0 REFERENCES

HNu Systems, Inc. Instruction Manual. Model PI 101, 1986.
HNu Systems, Inc. Operator's Manual. Model DL 101, 1991.

# F204
# *FLAME IONIZATION (FID)*

# FLAME IONIZATION DETECTOR (FID)
# FOXBORO OVA 128

## 1.0 PURPOSE

The purpose of this procedure is to provide general reference information for using the Foxboro OVA 128, Flame Ionization Detector (FID) or an equivalent or substitute device, in the field. Calibration, operation, and field maintenance will be included in this SOP. The OVA 128 is an intrinsically safe organic vapor monitor, but it cannot be used in atmospheres that are oxygen deficient and it is unable to detect inorganic compounds, including poisonous atmospheres.

## 2.0 SCOPE

This procedure provides information on the field operation and general maintenance of the Foxboro OVA 128. Application of the information contained herein will ensure that this type of field monitoring equipment will be properly operated. Review of the manufacturer's operating manual is a necessity for more detailed descriptions and operating information.

These procedures refer only to monitoring for health and safety. The methods are not directly applicable to surveillance of air quality for analytical purposes.

## 3.0 DEFINITIONS

FID - Flame Ionization Detector

ppm - parts per million: parts of vapor or gas per million parts of air (directly proportional to calibration gas).

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures where applicable, or that other approved procedures are developed. The Project Manager is responsible for selecting qualified individuals for the monitoring activities.

Project Health and Safety Officer (PHSO) - The Project Health and Safety Officer is responsible for developing a site-specific Health and Safety Plan (HASP) which specifies air monitoring requirements.

Field Team Leader - It is the responsibility of the Field Team Leader to implement these procedures in the field, and to ensure that the Field Investigation Personnel performing air monitoring activities have been briefed and trained to execute these procedures before the start of site operations.

Site Health and Safety Officer (SHSO) - The SHSO is responsible for ensuring that the specified air monitoring equipment is on site, calibrated, and used correctly by the Field Personnel. The SHSO will coordinate these activities with the Field Team Leader.

Field Investigation Personnel - It is the responsibility of the Field Investigation Personnel to follow these procedures or to follow documented project-specific procedures as directed by the Field Team Leader/Site

Health and Safety Officer.. The Field Investigation Personnel are responsible for documenting all air monitoring results in both the Field Logbook during each field investigation.

## 5.0 PROCEDURES

The following subsections will discuss calibration, operation and maintenance of the OVA 128. These sections, however, should not be used as a substitute for the manufacturer's operating manual.

The OVA 128 utilizes the principle of flame ionization whereby molecules enter the detector chamber and are exposed to a hydrogen flame which ionizes the organic vapors. As the vapors are burned they leave positively-charged carbon-containing ions. These positive ions are driven by an electric field to a collecting electrode. As the positive ions collect on the electrode, a current is created. This current corresponds to the collection rate of the positive ions. The current is then measured with a linear electrometer preamplifier which produces a signal proportional to the ionization current. This signal is then amplified by a signal conditioning amplifier and sent to the readout assembly and the strip chart recorder (when attached) in parts per million (ppm). The OVA 128 responds to virtually all organic compounds, that is, compounds that contain carbon-hydrogen or carbon-carbon bonds. The OVA 128 can operate in two modes:

Survey Mode: During normal survey mode operation, a sample is drawn into the probe and transferred to the detector chamber by an internal pumping system. When the sample reaches the FID, it is ionized as described above and the resulting signal is translated to the meter as a direct reading concentration for total organic vapors; or recorded as a quantifiable peak on a strip chart.

Gas Chromatography Mode: Gas Chromatography (GC) is a technique for separating components of a sample and qualitatively and quantitatively identifying them. This is possible when using the OVA128 GC Model. The sample to be separated is injected into a column packed with an inert solid. As the carrier gas (hydrogen) forces the sample through the column, the separated components of the sample are retained on the column for different periods of time. Each component will then be transferred to the detector chamber as described in the survey mode section. The response from each component will be recorded as a peak on the strip chart. The qualitative and quantitative results can then be determined only by qualified personnel.

### 5.1 Calibration

Primary calibration involves internal adjustments and should only be done by the manufacturer or an authorized equipment technician.

The following items will be needed for secondary calibration of the OVA 128:

- Calibration gas (usually methane but can be contaminant specific).
- Gas regulator.
- Tedlar bag (usually two to three liter capacity).
- Tubing.
- Screw driver set.
- Flame Ionization Detector (FID) Calibration Form.

Points to follow for "Single Sample Calibration:"

- Follow operation procedures up to "calibration" step.

- For methane calibration in the 90 to 100 ppm range, set scale to x10 and gas select control to 300.

- Use calibration adjustment knob to "zero" the meter. Care must be taken to document background readings before zeroing the instruments.

- Introduce methane sample of known concentration from Tedlar Bag and adjust gas select knob so that meter reading equals sample concentration. Withdraw methane sample, allow meter reading to stabilize and repeat. Discontinue calibration when meter reaction to sample introduction is consistent.

- If there is a problem with calibration, consult the manufacturer's operating manual or an experienced technician.

- Refer to manufacturer's operating manual for additional information.

Since the OVA 128 uses flame ionization, it has a broad application in terms of which organic contaminants it can ionize. Note: the OVA 128 hydrogen flame ionization detector is more sensitive to hydrocarbons than any other class of organic compounds.

## 5.2 Operation

Review of the Startup Procedure Chapter of the manufacturer's operating manual (page 7), is critical prior to actual field operation.

Points to follow for startup of the OVA 128:

- After the OVA 128 is assembled check that the battery level is sufficient for operation. A minimum, reading of 7.5 on the x1 scale should provide four hours of operation.

- Turn instrument switch on and allow a minimum of a five minute warmup before turning pump on.

- Turn the pump switch on. Pump operation is audible.

- Place the instrument panel in the vertical position and check the sample flow rate indicator. The normal range is 1.5 to 2.0 units. If less, filters may need to be changed, or the battery may need further charging or replacement.

- Ensure that an air-tight seal exists from the probe back to the instrument by placing your thumb over the end of the probe. Cover the probe long enough to shut the pump down (approximately 5-10 seconds). If the pump does not shut down, check all fittings and connections from the probe assembly back to the instrument. An air-tight seal is very important for obtaining an accurate reading. Readings obtained without an air-tight seal are diluted and not indicative of actual concentrations (survey mode) or concentrations and contaminants present (GC mode).

- Use calibrate adjust knob to set meter to predetermined level for activating audible alarm (if desired).

- Set to x1 scale and adjust meter reading to zero.

- Open hydrogen tank valve to be certain that there is enough hydrogen in the tank for operation approximately 1.5 to 2 full turns. The OVA 128 will use approximately 150 psi/hour. Then open the hydrogen supply valve approximately 1.5 to 2 full turns noting that the gauge reading should be within the range of 8 to 12 psi.

Note: Caution must be used when filling the hydrogen tank to maintain safe operating pressures and temperatures. Only prepurified or zero grade hydrogen will be used. Use the hydrogen filling hose supplied by the manufacturer, and hydrogen tank with a rated pressure that does not exceed the capacity of the fill line.

- After approximately one minute depress the igniter button until the hydrogen has been ignited (needle on readout assembly should deflect to full-scale). DO NOT HOLD THE IGNITER BUTTON IN FOR MORE THAN FIVE SECONDS. If hydrogen does not light, wait two minutes and try again. If it still does not light, consult manufacturer's operating manual.

  Note: Hydrogen gas will not ignite if battery is too low.

- The instrument is now ready for calibration, if required.

Once the OVA 128 has been running and stabilized for 15 minutes, it is ready for "Survey Mode" procedures. Set the calibrate switch to the desired range and the OVA 128 is now ready for field operation.

Note: Care must be taken when operating the OVA 128. Special areas of concern are the probe assembly and the analyzer. Do not stick the probe in water or soil; this will give erroneous readings and could possibly damage the pump. The analyzer unit must be kept clean and away from physical hazards, and the exhaust free from obstructions.

For shutdown and refueling, follow manufacturer's recommend procedures.

## 5.3 Maintenance

Preventive maintenance consists of keeping the Foxboro OVA 128 as clean as possible. The OVA 128 must be decontaminated and wiped down with a damp cloth after each use.

The other type of maintenance is the manufacturer's scheduled maintenance which consists of the following:

- Check particle filters on a monthly basis.
- Check quad rings on a monthly basis or as needed.
- Clean burner chamber on a quarterly basis or as needed.
- Primary calibration and factory check on an annual basis or when non-operational.
- Secondary calibration on a daily or weekly basis depending on usage.

### 5.4 Shipping

Following is information regarding the transport of FID meters and calibration gas.

- Compressed gas cylinders (i.e., methane and hydrogen) may not be carried on aircraft. These cylinders should either be shipped to the jobsite, or purchased locally.

- Shipping of the gas cylinders requires completion of a form (specified by the shipping company), that identifies the container as compressed gas. Compressed gas stickers must be affixed to the package.

## 6.0 QUALITY ASSURANCE RECORDS

Quality assurance records will be maintained for each air monitoring event. The following information shall be recorded in the Field Logbook.

- Identification - Site name, location, project and task number, activity monitored, (surface water sampling, soil sampling, etc.), serial number, time, resulting concentration, comments and identity of air monitoring personnel.

- Field observations - Appearance of sampled media (if definable).

- Additional remarks (e.g., the OVA 128 meter had wide range fluctuations during air monitoring activities).

## 7.0 REFERENCES

Foxboro Model OVA 128 Century Organic Vapor Analyzer Instruction Manual, 1985.

# F301
# *SAMPLE PRESERVATION AND HANDLING*

# SAMPLE PRESERVATION AND HANDLING

## 1.0 PURPOSE

This SOP describes the appropriate containers for samples of particular matrices, and the steps necessary to preserve those samples when shipped off site for chemical analysis. It also identifies the qualifications for individuals responsible for the transportation of hazardous materials and samples and the regulations set forth by the Department of Transportation regarding the same.

## 2.0 SCOPE

Some chemicals react with sample containers made of certain materials; for example, trace metals adsorb more strongly to glass than to plastic, while many organic chemicals may dissolve various types of plastic containers. It is therefore critical to select the correct container in order to maintain the integrity of the sample prior to analysis.

Many water and soil samples are unstable and may change in chemical character during shipment. Therefore, preservation of the sample may be necessary when the time interval between field collection and laboratory analysis is long enough to produce changes in either the concentration or the physical condition of the constituent(s). While complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological changes that may occur after the sample is collected.

Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/freezing. Their purposes are to (1) retard biological activity, (2) retard hydrolysis of chemical compounds/complexes, (3) reduce constituent volatility, and (4) reduce adsorption effects.

Typical sample container and preservation requirements for this project are provided in Attachment A of this SOP. Note that sample container requirements (i.e., volumes) may vary by laboratory.

The Department of Transportation, Code of Federal Regulations (CFR) Title 49 establishes regulations for all materials offered for transportation. The transportation of environmental samples for analysis is regulated by Code of Federal Regulations Title 40 (Protection of the Environment), along with 49 CFR Part 172 Subpart H. The transportation of chemicals used as preservatives and samples identified as hazardous (as defined by 49 CFR Part 171.8) are regulated by 49 CFR Part 172.

## 3.0 DEFINITIONS

HCl - Hydrochloric Acid

$H_2SO_4$ - Sulfuric Acid

$HNO_3$ - Nitric Acid

NaOH - Sodium Hydroxide

Normality (N) - Concentration of a solution expressed as equivalents per liter, where an equivalent is the amount of a substance containing one mole of replaceable hydrogen or its equivalent. Thus, a one molar

solution of HCl, containing one mole of H, is "one-normal," while a one molar solution of $H_2SO_4$ containing two moles of H, is "two-normal."

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other, approved procedures are developed. The Project Manager is responsible for development of documentation of procedures which deviate from those presented herein. The Project Manager is also responsible for proper certification of individuals responsible for transportation of samples of hazardous substances.

Field Team Leader - It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures. The Field Team Leader is responsible to ensure all samples and/or hazardous substances are properly identified, labeled, and packaged prior to transportation.

Sampling Personnel - It is the responsibility of the field sampling personnel to initiate sample preservation and handling. It is also the responsibility of the field sampling personnel to understand and adhere to the requirements for proper transportation of samples and/or hazardous substances.

## 5.0 PROCEDURES

The following procedures discuss sample containerization and preservation techniques that are to be followed when collecting environmental samples for laboratory analysis.

### 5.1 Sample Containers

For most samples and analytical parameters either glass or plastic containers are satisfactory. In general, if the analyte(s) to be measured is organic in nature, the container shall be made of glass. If the analyte(s) is inorganic, then glass or plastic containers may be used. Containers shall be kept out of direct sunlight (to minimize biological or photo-oxidation/photolysis of constituents) until they reach the analytical laboratory. The sample container shall have approximately five to ten percent air space ("ullage") to allow for expansion/vaporization if the sample is heated during transport (one liter of water at 4°C expands by 15 milliliters if heated to 130°F/55°C); however, head space for volatile organic analyses shall be omitted.

The analytical laboratory shall provide sample containers that have been certified clean according to USEPA procedures. Shipping containers for samples, consisting of sturdy ice chests, are to be provided by the laboratory.

Once opened, the sample container must be used at once for storage of a particular sample. Unused, but opened, containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or missing liners (if required for the container) shall be discarded.

General sample container, preservative, and holding time requirements are listed in Attachment A.

## 5.2 Preservation Techniques

The preservation techniques to be used for various analytes are listed in Attachment A. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or added in the field using laboratory supplied preservatives. Some of the more commonly used sample preservation techniques include storage of sample at a temperature of 4°C, acidification of water samples, and storage of samples in dark (i.e. amber) containers to prevent the samples from being exposed to light.

All samples shall be stored at a temperature of 4°C. Additional preservation techniques shall be applied to water samples as follows:

- Water samples to be analyzed for volatile organics shall be acidified.
- Water samples to be analyzed for semivolatile organics shall be stored in dark containers.
- Water samples to be analyzed for pesticides/PCBs shall be stored in dark containers.
- Water samples to be analyzed for inorganic compounds shall be acidified.

These preservation techniques generally apply to samples of low-level contamination. The preservation techniques utilized for samples may vary. However, unless documented otherwise in the project plans, all samples shall be considered low concentration. All samples preserved with chemicals shall be clearly identified by indicating on the sample label that the sample is preserved.

## 5.3 Sample Holding Times

The elapsed time between sample collection and initiation of laboratory analyses is considered the holding time and must be within a prescribed time frame for each individual analysis to be performed. Sample holding times for routine sample collection are provided in Attachment A.

# 6.0 SAMPLE HANDLING AND TRANSPORTATION

After collection, the outside of all sample containers will be wiped clean with a damp paper towel; however sample handling should be minimized. Personnel should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, personnel should ensure that melted ice cannot cause sample containers to become submerged, as this may result in sample cross-contamination and loss of sample labels. Sealable plastic bags, (zipper-type bags), should be used when glass sample containers are placed in ice chests to prevent cross-contamination, if breakage should occur.

Samples may be hand delivered to the laboratory or they may be shipped by common carrier. Relevant regulations for the storage and shipping of samples are contained in 40 CFR 261.4(d). Parallel state regulations may also be relevant. Shipment of dangerous goods by air cargo is also regulated by the United Nations/International Civil Aviation Organization (UN/ICAO). The Dangerous Goods Regulations promulgated by the International Air Transport Association (IATA) meet or exceed DOT and UN/ICAO requirements and should be used for shipment of dangerous goods via air cargo. Standard procedures for shipping environmental samples are given in Attachment B.

## 7.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater. 15th Edition. APHA, Washington, D.C.

USEPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

USEPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020. USEPA EMSL, Cincinnati, Ohio.

USEPA, Region IV, 1991. Environmental Compliance Branch Standard Operating Procedures and Quality Assurance Manual. Athens, Georgia.

Protection of the Environment, Code of Federal Regulation, Title 40, Parts 260 to 299.

Transportation, Code of Federal Regulation, Title 49, Parts 100 to 177.

# ATTACHMENT A

## REQUIRED CONTAINER, PRESERVATION TECHNIQUES AND HOLDING TIMES

ATTACHMENT A

## SUMMARY OF CONTAINERS, PRESERVATION, AND HOLDING TIMES FOR AQUEOUS SAMPLES

| Parameter | Bottle Requirements | Preservation Requirements | Holding Time [1] | Analytical Method | Bottle Volume |
|---|---|---|---|---|---|
| Volatile Organic Compounds (VOA) | glass teflon lined cap | Cool to 4°C<br>1:1 HCl pH <2 | 14 days | CLP | 2 x 40 ml |
| Semivolatile Organic Compounds (SVOA) | glass teflon lined cap | Cool to 4°C<br>Dark | Extraction within 5 days<br>Analyze 40 days | CLP | 2 x 1 liter |
| PCB/Pesticides | glass teflon lined cap | Cool to 4°C<br>Dark | Extraction within 5 days<br>Analyze 40 days | CLP | 2 x 1 liter |
| Cyanide | plastic/glass | NaOH to pH>12<br>Cool to 4°C | 14 days | CLP<br>EPA 335.2 | 1 x 1 liter |
| Metals (TAL) | plastic/glass | $HNO_3$ to pH <2 | 180 days except Mercury is 26 days | CLP | 1 x 1 liter |
| Total Organic Carbon | glass, teflon lined cap | Cool to 4°C<br>$H_2SO_4$ to pH <2 | 28 days | EPA 415.1 | 2 x 40 ml |
| Total Organic Halogen | plastic/glass | Cool to 4°C<br>$H_2SO_4$ to pH <2 | 28 days | EPA 450.1 | 250 ml |
| Chloride | plastic/glass | none required | 28 days | EPA 325.2/325.3 | 250 ml |
| Sulfate | plastic/glass | Cool to 4°C | 28 days | EPA 375.4 | 250 ml |
| Alkalinity | plastic/glass | Cool to 4°C | 14 days | EPA 310.1/310.2 | 250 ml |
| Gross alpha/gross beta | plastic/glass | $HNO_3$ to pH <2 | 6 months | 9310 | 1 gallon |
| Chlorinated herbicides | glass, teflon lined cap | Cool to 4°C | 14/28 days | EPA 515.1 | 1000 ml |
| Hardness | plastic/glass | $HNO_3$ to pH <2 | 6 months | EPA 130.2 | 150 ml |

[1] Holding times for CLP methods are based on Validated Time of Sample Receipt as stated in CLP statement of work of February, 1991. Holding times for Non-CLP methods are based on time of sample collection.

Note: Verify this information with the laboratory that will perform the analyses.

ATTACHMENT A (Continued)

SUMMARY OF CONTAINERS, PRESERVATION, AND HOLDING TIMES FOR SOIL SAMPLES

| Parameter | Bottle Requirements | Preservation Requirements | Holding Time [1] | Analytical Method | Bottle Volume |
|---|---|---|---|---|---|
| Volatile Organic Compounds (VOA) | 3 Encore™ samplers or glass jar with Teflon-lined septum | Cool to 4°C | 14 days | CLP | 1 x 6 ml[2] |
| Semivolatile Organic Compounds (SVOA) | glass teflon lined cap | Cool to 4°C | Extraction within 10 days<br>Analyze 40 days | CLP | 1 x 250 gm |
| PCB/Pesticides | glass teflon lined cap | Cool to 4°C | Extraction within 10 days<br>Analyze 40 days | CLP | 1 x 50 gm |
| Metals (TAL) | plastic/glass | Cool to 4°C | Mercury is 26 days<br>180 days | CLP | 1 x 50 gm |
| Cyanide | plastic/glass | Cool to 4°C | 14 days | CLP<br>EPA 335.2M | 1 x 50 gm |

(1) Holding times for CLP methods are based on Validated Time of Sample Receipt as stated in CLP statement of work of February, 1991. Holding times for Non-CLP methods are based on time of sample collection.

(2) To be used if Encore™ samplers are not applicable.

Note: Verify this information with the laboratory that will perform the analyses.

# ATTACHMENT B

# SAMPLE SHIPPING PROCEDURES

## ATTACHMENT B

## SAMPLE SHIPPING PROCEDURES

### Introduction

Samples collected during field investigations or in response to a hazardous materials incident must be classified by the project leader, prior to shipping by air, as either environmental or hazardous substances. The guidance for complying with U.S. DOT regulations in shipping environmental laboratory samples is given in the "National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."

Pertinent regulations for the shipping of environmental samples is given in 40 CFR 261.4(d). Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated may require shipment as dangerous goods/hazardous substance. Regulations for packing, marking, labeling, and shipping of dangerous goods by air transport are promulgated by the United Nations International Civil Aviation Organization (UN/ICAO),which is equivalent to IATA.

Individuals responsible for transportation of environmental samples or dangerous goods/hazardous substances must be tested and certified by their employer. This is required by 49 CFR Part 172 Subpart H Docket HM-126 to assure the required qualifications for individuals offering materials for transportation.

Environmental samples shall be packed prior to shipment by commercial air carrier using the following procedures:

1. Select a sturdy cooler in good repair. Secure and tape the drain plug (inside and outside) with fiber or duct tape. Line the cooler with a large heavy duty plastic bag. This practice keeps the inside of the cooler clean and minimizes cleanup at the laboratory after samples are removed.

2. Allow sufficient headspace (ullage) in all bottles (except VOAs) to compensate for any pressure and temperature changes (approximately 10 percent of the volume of the container).

3. Be sure the lids on all bottles are tight (will not leak). In many regions custody seals are also applied to sample container lids. The reason for this practice is two-fold: to maintain integrity of samples and keep lid on the container should the lid loosen during shipment. Check with the appropriate regional procedures prior to field work. In many cases, the laboratory manager of the analytical lot to be used on a particular project can also provide this information.

4. It is good practice to wrap all glass containers in bubblewrap or other suitable packing material prior to placing in plastic bags.

5. Place all bottles in separate and appropriately sized polyethylene bags and seal the bags with tape (preferably plastic electrical tape, unless the bag is a zipper-type bag). Up to three VOA bottles, separately wrapped in bubblewrap, may be packed in one plastic bag.

6. Optionally, place three to six VOA vials in a quart metal can and then fill the can with vermiculite.

7. Place two to four inches of vermiculite (ground corn cob, or other inert packing material) in the bottom of the cooler and then place the bottles and cans in the cooler with sufficient space to allow for the addition of more vermiculite between the bottles and cans.

8. Put frozen "blue ice" (or ice that has been placed in properly sealed, double-bagged, heavy duty polyethylene bags) on top of and between the samples. Fill all remaining space between the bottles or cans with packing material. Fold and securely fasten the top of the large heavy duty plastic bag with tape (preferably electrical or duct).

9. Place the Chain-of-Custody Record and the Request for Analysis Form (if applicable) into a plastic bag, tape the bag to the inner side of the cooler lid, and then close the cooler and securely tape (preferably with fiber tape) the top of the cooler unit. Wrap the tape three to four times around each side of the cooler unit. Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

10. Each cooler (if multiple coolers) should have its own Chain-of-Custody Record reflecting the samples shipped in that cooler.

11. Label according to 40 CFR 261.4(d). The shipping containers should be marked "THIS END UP," and arrow labels which indicate the proper upward position of the container should be affixed to the container. A label containing the name and address of the shipper and laboratory shall be placed on the outside of the container. It is good practice to secure this label with clear plastic tape to prevent removal during shipment by blurring of important information should the label become wet. The commercial carrier is not required to sign the COC record as long as the custody seals remain intact and the COC record stays in the cooler. The only other documentation required is the completed airbill, which is secured to the top of the shipping container. Please note several coolers/shipping containers may be shipped under one airbill. However, each cooler must be labeled as "Cooler 1 of 3, Cooler 2 of 3, etc.", prior to shipping. Additionally it is good practice to label each COC form to correspond to each cooler (i.e., 1 of 3, 2 of 3, etc.).

# F302
# *CHAIN-OF-CUSTODY*

# CHAIN-OF-CUSTODY

## 1.0 PURPOSE

The purpose of this SOP is to provide information on chain-of-custody procedures to be used to document sample handling.

## 2.0 SCOPE

This procedure describes the steps necessary for transferring samples through the use of Chain-of-Custody Records. A Chain-of-Custody Record is required, without exception, for the tracking and recording of samples collected for on-site or off-site analysis (chemical or geotechnical) during program activities (except wellhead samples taken for measurement of field parameters). Use of the Chain-of-Custody Record Form creates an accurate written record that can be used to trace the possession and handling of the sample from the moment of its collection through analysis. This procedure identifies the necessary custody records and describes their completion. This procedure does not take precedence over region-specific or site-specific requirements for chain-of-custody.

## 3.0 DEFINITIONS

Chain-of-Custody Record Form - A Chain-of-Custody Record Form is a printed two-part form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to another custodian. One copy of the form must be retained in the project file. An example of a Chain-of-Custody Record Form is presented in Attachment B.

Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under one's custody if:

- It is in one's actual possession.
- It is in one's view, after being in one's physical possession.
- It was in one's physical possession and then he/she locked it up to prevent tampering.
- It is in a designated and identified secure area.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other, approved procedures are developed. The Project Manager is responsible for development of documentation of procedures which deviate from those presented herein. The Project Manager is responsible for ensuring that chain-of-custody procedures are implemented. The Project Manager also is responsible for determining that custody procedures have been met by the analytical laboratory.

Field Team Leader - The Field Team Leader is responsible for determining that chain-of-custody procedures are implemented up to and including release to the shipper or laboratory. It is the responsibility

of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures.

Sampling Personnel - It is the responsibility of the field sampling personnel to initiate chain-of-custody procedures, and maintain custody of samples until they are relinquished to another custodian, the sample shipper, or to a common carrier.

## 5.0 PROCEDURES

The term "chain-of-custody" refers to procedures which ensure that evidence presented in a court of law is valid. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the courtroom, as well as providing security for the evidence as it is moved and/or passed from the custody of one individual to another.

Chain-of-custody procedures, record keeping, and documentation are an important part of the management control of samples. Regulatory agencies must be able to provide the chain-of-possession and custody of any samples that are offered for evidence, or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

### 5.1 Sample Identification

The method of identification of a sample depends on the type of measurement or analysis performed. When in-situ measurements are made, the data are recorded directly in bound logbooks or other field data records with identifying information.

Information which shall be recorded in the Field Logbook, when in-situ measurements or samples for laboratory analysis are collected, includes:

- Field Sampler(s)
- Project Number
- Project Sample Number
- Sample location or sampling station number, if applicable
- Date and time of sample collection and/or measurement
- Field observations
- Equipment used to collect samples and measurements
- Calibration data for equipment used

Measurements and observations shall be recorded using waterproof ink.

### 5.1.1 Sample Label

Samples, other than in-situ measurements, are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often divided into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the Sampling and Analysis Plan. Each sample container is identified by a sample label (see Attachment A). Sample labels are provided, along with sample containers, by the analytical laboratory. The information recorded on the sample label includes:

- Project Number.
- Station Location - The unique sample number identifying this sample.
- Date - A six-digit number indicating the day, month, and year of sample collection (e.g., 12/21/85).
- Time - A four-digit number indicating the 24-hour time of collection (for example: 0954 is 9:54 am., and 1629 is 4:29 p.m.).
- Medium - Water, soil, sediment, sludge, waste, etc.
- Sample Type - Grab or composite.
- Preservation - Type and quantity of preservation added.
- Analysis - VOA, BNAs, PCBs, pesticides, metals, cyanide, other.
- Sampled By - Printed name of the sampler.
- Remarks - Any pertinent additional information.

Using only the work assignment number of the sample label maintains the anonymity of sites. This may be necessary, even to the extent of preventing the laboratory performing the analysis from knowing the identity of the site (e.g., if the laboratory is part of an organization that has performed previous work on the site).

## 5.2 Chain-of-Custody Procedures

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

### 5.2.1 Field Custody Procedures

- Samples are collected as described in the site Sampling and Analysis Plan. Care must be taken to record precisely the sample location and to ensure that the sample number on the label matches the Chain-of-Custody Record exactly.

- The person undertaking the actual sampling in the field is responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

- When photographs are taken of the sampling as part of the documentation procedure, the name of the photographer, date, time, site location, and site description are entered sequentially in the Field Logbook as photos are taken. Once developed, the photographic prints shall be serially numbered, corresponding to the logbook descriptions; photographs will be stored in the project files. It is good practice to identify sample locations in photographs by including an easily read sign with the appropriate sample/location number.

- Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions, e.g., a logbook notation would explain that a pencil was used to fill out the sample label if the pen would not function in freezing weather.

### 5.2.2 Transfer of Custody and Shipment

Samples are accompanied by a Chain-of-Custody Record Form. When transferring the possession of samples, the individual(s) relinquishing and receiving will sign, date, and note the time on the Record. This Record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. The Chain-of-Custody Record is filled out as given below.

- Enter header information (Project and Task number, samplers, and project name).

- Enter sample specific information (sample number, media, sample analysis required and analytical method, grab or composite, number and type of sample containers, and date/time sample was collected).

- Sign, date, and enter the time under "Relinquished by" entry.

- Have the person receiving the sample sign the "Received by" entry. If shipping samples by a common carrier, print the carrier to be used in this space (i.e., Federal Express).

- If a carrier is used, enter the airbill number under "Remarks," in the bottom right corner.

- Place the original (top, signed copy) of the Chain-of-Custody Record Form in a plastic zipper-type bag or other appropriate sample shipping package. Retain the copy with field records.

- Sign and date the custody seal, a 1 - by 3-inch white paper label with black lettering and an adhesive backing. Attachment C is an example of a custody seal. The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field. Custody seals shall be provided by the analytical laboratory.

- Place the seal across the shipping container opening so that it would be broken if the container was to be opened.

- Complete other carrier-required shipping papers.

The custody record is completed using waterproof ink. Any corrections are made by drawing a line through and initialing and dating the change, then entering the correct information. Erasures are not permitted.

Common carriers will usually not accept responsibility for handling Chain-of-Custody Record Forms; this necessitates packing the record in the shipping container (enclosed with other documentation in a plastic zipper-type bag). As long as custody forms are sealed inside the shipping container and the custody seals are intact, commercial carriers are not required to sign the custody form.

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Record, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis.

## 6.0 QUALITY ASSURANCE RECORDS

Once samples have been packaged and shipped, the COC copy and airbill receipt becomes part of the Quality Assurance Record.

## 7.0 REFERENCES

1. USEPA. User's Guide to the Contract Laboratory Program. Office of Emergency and Remedial Response, Washington, D.C. (EPA/540/P-91/002), January 1991.

ATTACHMENT A

EXAMPLE CLIENT (SAMPLE) LABEL

## CLIENT LABEL

Client: _____ Date: _____

Site: _____ Time: _____

Sample ID: _____

Analysis: _____

Signature: _____

OUA-8022 (10/98)

# ATTACHMENT B

# EXAMPLE CHAIN-OF-CUSTODY RECORD

# Cus. / Record

OUA-4124

| Client | Project Manager | Date | Chain Of Custody Number 618 |
|---|---|---|---|
| Address | Telephone Number (Area Code)/Fax Number | Lab Number | Page ____ of ____ |
| City / State / Zip Code | Site Contact | | |
| Project Name | Carrier/Waybill Number | | |
| Contract/Purchase Order/Quote No. | | | |

| Sample I.D. No. and Description | Date | Time | Sample Type | Total Volume | Containers Type | Containers No. | Preservative | Condition on Receipt | Analysis |
|---|---|---|---|---|---|---|---|---|---|
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Special Instructions

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For ____ Months

Turn Around Time Required
☐ Normal ☐ Rush

QC Level
☐ I. ☐ II. ☐ III.

Project Specific (Specify)

| | Date | Time | | Date | Time |
|---|---|---|---|---|---|
| 1. Relinquished By | | | 1. Received By | | |
| 2. Relinquished By | | | 2. Received By | | |
| 3. Relinquished By | | | 3. Received By | | |

Comments

# ATTACHMENT C

# EXAMPLE CUSTODY SEAL

QUA-145

Quanterra

No

Custody Seal

DATE

SIGNATURE

Quanterra

No

# F303
# *FIELD LOGBOOK*

# FIELD LOGBOOK FIELD LOGBOOK

## 1.0 PURPOSE

This SOP describes the process for maintaining a Field Logbook.

## 2.0 SCOPE

The Field Logbook is a document which records all major on-site activities conducted during a field investigation. At a minimum, the following activities/events shall be recorded in the Field Logbook by each member of the field crew.

- Arrival/departure of site workers and visitors
- Arrival/departure of equipment
- Sample pickup (sample numbers, carrier, time)
- Sampling activities
- Start or completion of boreholes, monitoring wells, or sampling activities
- Health and safety issues

The Field Logbook is initiated upon arrival at the site for the start of the first on-site activity. Entries are made every day that on-site activities take place. At least one field logbook shall be maintained per site.

The Field Logbook becomes part of the permanent site file. Because information contained in the Field Logbook may be admitted as evidence in legal proceedings, it is critical that this document is properly maintained.

## 3.0 DEFINITIONS

Field Logbook - The Field Logbook is a bound notebook with consecutively numbered pages. Upon entry of data, the logbook requires the signature of the responsible data/information recorder.

## 4.0 RESPONSIBILITIES

The Field Team Leader is responsible for maintaining a master field logbook for the duration of on-site activities. Each member of the sampling crew is responsible for maintaining a complete and accurate record of site activities for the duration of the crew members participation in the project.

## 5.0 PROCEDURES

The following sections present some of the information that must be recorded in the Field Logbook. In general, a record of all events and activities, as well as other potentially important information shall be recorded by each member of the field team.

## 5.1 Cover

The inside cover or title page of each field logbook shall contain the following information:

- Project Number
- Project name and location
- Name of Field Team Leader
- Baker's address and telephone number
- Start date
- If several logbooks are required, a sequential Field Logbook number

It is good practice to list important phone numbers and points of contact here.

## 5.2 Daily Entries

Daily entries into the logbook may contain a variety of information. At the beginning of each day the following information must be recorded by each team member.

- Date
- Start time
- Weather
- All field personnel present
- All visitors present
- Other pertinent information (i.e., planned activities, schedule changes, expected visitors, and equipment changes)

During the day, an ongoing record of all site activities should be written in the logbook. The master logbook kept by the field team leader need not duplicate that recorded in other Field Logbooks, but should summarize the information in other books and, where appropriate, reference the page numbers of other logbooks where detailed information pertaining to a subject may be found.

Some specific information which must be recorded in the logbook includes the following:

- Equipment used, equipment numbers, calibration, field servicing
- Field measurements
- Sample numbers, media, bottle size, preservatives, collection methods, and time
- Test boring and monitoring well construction information, including boring/well number and location
- Sketches for each sample location including appropriate measurements if required
- Photograph log
- Drum log
- Other pertinent information

All entries should be made in indelible ink; all pages numbered consecutively; and all pages must be signed or initialed and dated by the responsible field personnel completing the log. No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single line, initialed, and dated.

### 5.3 Photographs

If photographs are permitted at the site, the record shall be maintained in the Field Logbook. When movies, slides or photographs are taken of any site location, they are numbered or cross-referenced to correspond to logbook entries. The name of the photographer, date, time, site location, site description, direction of view and weather conditions are entered in the logbook as the photographs are taken. Special lenses, film, or other image-enhancement techniques also must be noted in the Field Logbook. Once processed, photographs shall be serially numbered and labeled corresponding to the Field Logbook entries.

## 6.0 QUALITY ASSURANCE RECORDS

Once on-site activities have been completed, the Field Logbook shall be considered a quality assurance record.

## 7.0 REFERENCES

None.

# F304
# *QA/QC SAMPLES*

# QUALITY CONTROL SAMPLES

## 1.0 PURPOSE

The SOP describes the type and quantity of Quality Control (QC) samples to be collected for most field sampling operations.

## 2.0 SCOPE

QC samples are those samples (usually collected in the field) that are sent to the laboratory along with the environmental samples in order to evaluate site conditions and laboratory precision and accuracy. Evaluation of the results from the QC samples allows for the quality of the data to be assessed. There are five different type of QC samples: trip blanks, equipment rinsate blanks, field blanks, duplicates and matrix spike/matrix spike duplicate (MS/MSD) samples. The first three types of QC samples are used to assess field conditions during sampling and/or transport of the environmental samples. The latter two types of QC samples are used by the laboratory to help assess precision and accuracy. (The laboratory also has other internal samples and procedures to assess precision and accuracy.)

Depending on the level of data quality required by the project, different amounts of QC samples are collected. These are described in detail below.

## 3.0 DEFINITIONS

Trip Blank - Trip blanks are 40-ml volatile organic analysis (VOA) vials of ASTM Type II water that are filled at the laboratory, transported to the sampling site, and returned to the laboratory with environmental VOA samples. Trip blanks are not opened in the field.

Equipment Rinsates - Equipment rinsates are samples of ASTM Type II water (provided by the laboratory) passed over decontaminated sampling equipment. They are used as a measure of the effectiveness of the decontamination procedure. The rinsate is analyzed for the same parameters as the environmental samples collected from the piece of equipment.

Field Blanks - Field blanks are samples of source water used for decontamination and steam cleaning. At a minimum there is one sample collected for each source of water used per sampling event. The field blank is analyzed for all the parameters tested during the sampling event.

Duplicates - Duplicates are collected to help assess laboratory reproducibility (precision). Solid matrix samples are homogenized before being split, except for VOAs, which are not homogenized because of potential volatile loss. Liquid matrix samples are collected simultaneously. For both solid and liquid matrices, double the normal volume is required. The same analyses are completed on the duplicate as on the environmental sample.

MS/MSD - MS/MSD samples are used by the laboratory (but collected in the field) to help determine both precision and accuracy of analysis. For liquid matrices, triple the volume of sample is required (that is, one volume for the environmental sample, one volume for the MS sample, and one volume for the MSD sample). For solid matrices, additional volume is usually not required (although this will depend upon the laboratory).

MS/MSD - MS/MSD samples are used by the laboratory (but collected in the field) to help determine both precision and accuracy of analysis. For liquid matrices, triple the volume of sample is required (that is, one volume for the environmental sample, one volume for the MS sample, and one volume for the MSD sample). For solid matrices, additional volume is usually not required (although this will depend upon the laboratory).

## 4.0 RESPONSIBILITIES

The Project Manager is responsible for estimating the number of QC samples required for any sampling event. The procedures for determining the number is described in Section 5.0 below. The Field Team Leader is responsible for making sure that the sampling team(s) are collecting the required number of QC samples. The Field Team member(s) are responsible for understanding the rationale and methods for QC sample collection and for coordinating QC sample collection as appropriate.

## 5.0 PROCEDURES

The procedures for QC sample collection and the frequency at which each type of sample should be collected is described below.

### 5.1 Trip Blanks

Trip blanks (one pair of 40 ml vials) are sent to the laboratory in each cooler which contains samples for volatile organic analyses. The trip blank should also be kept in the field, with the volatile samples, during the period of sample collection. Analyses of the trip blank will determine if the sample containers were contaminated prior to sampling or during transport.

### 5.2 Equipment Rinsates

Equipment rinsates are collected by pouring analyte-free water (provided by the laboratory) over decontaminated sampling equipment and collecting the rinsate. These are collected at a frequency of once per 10 samples and are analyzed for the same parameters as are the samples collected from that equipment. If two (or more) different types of equipment are used to collect samples in the same day (say by two field teams, one collecting soil samples from split spoons and one collecting groundwater from bailers), then two separate rinsate samples may be collected. The rinsate blank is used to qualify data.

### 5.3 Field Blanks

One field blank per source of water used for decontamination per sampling event is collected for all the parameters analyzed during that sampling event. In general, two field blanks are collected - one from the potable water source used for steam cleaning and one from distilled water purchased at a local store for use in general decontamination. The field blank is collected by opening up the water source at the sampling locations and pouring the water directly into the appropriate sample bottles. Analysis of the sample will indicate whether contamination was introduced into the samples during the collection process.

### 5.4 Field Duplicates

Field duplicates are collected at a frequency of 10 percent (one duplicate or per 10 samples) for levels C and D analyses, and at 5 percent (or one duplicate per 20 samples) for Level E analyses. The samples are split as described above and in other SOPs related to sample collection procedures. The number of duplicates to collect for levels C and D analyses is determined as follows: 1-10 environmental samples, 1 duplicate; 11-20 environmental samples, 2 duplicates; 21-30 environmental samples, 3 duplicates, etc. Field duplicates are useful in documenting the precision of the sampling process.

### 5.5 MS/MSD

MS/MSD samples are collected in the same manner as for a duplicate sample, except that triple the volume is required for analysis (for liquids). Generally no additional volume is required by the laboratory for solid samples (confirm with A2l). The frequency of collection is one MS/MSD pair (or two additional sample volumes) for each 20 environmental samples collected of similar matrix (e.g. groundwater, surface water, soil). The number of MS/MSD samples to collect is determined as follows: 1-20 environmental samples, one MS/MSD pair; 21-40 environmental samples, two MS/MSD pairs, etc.

## 6.0 SAMPLE COLLECTION RECORDS AND EVALUATION

Records of collection of QC samples are kept in the field logbooks and on the Chain-of-Custody forms. Evaluation of the results from the QC samples is performed by the laboratory and through data validation for the MS/MSD samples. Results of the other QC samples are compared to analytical results from the sampling event to determine if any field procedures, or sample transportation/handling may have adversely affected the concentrations found in the environmental samples.

## 7.0 REFERENCES

Hazardous Waste Remedial Actions Program. 1990. Requirements for Quality Control of Analytical Data. DOE/HWP-65/R1, US Department of Energy, Oak Ridge, Tennessee.

USEPA, 1988. User's Guide to the Contract Laboratory Program. 9240.0-1, Office of Emergency and Remedial Response, Washington, D.C.

USEPA, 1990. Quality Assurance/Quality Control Guidance for Removal Activities - Sampling QA/QC Plan and Data Validation Procedures (Interim Final). EPA/540/G-90/004, Office of Emergency and Remedial Response, Washington, D.C.

# F401
# *AQUIFER TESTING*

# AQUIFER TESTING (PUMP-TESTS)

## 1.0 PURPOSE

This SOP provides a general description of the technical methods and field procedures of a representative suite of aquifer testing (pump-tests) to calculate the aquifer parameters. Well-head testing (slug tests) to approximate part of these parameters is discussed in SOP F402. The descriptions herein are general in nature and do not apply to a specific well, well-field or project. Prior to designing pump-tests as part of a site investigation and during execution of the tests, the Project Manager, Site Manager and Program Geohydrologist must consult on the appropriate procedures; these procedures must then be recorded in the project documents.

## 2.0 SCOPE

The aquifer tests apply both to consolidated and unconsolidated strata; and to confined, semiconfined and phreatic conditions. The aquifer parameters subject to evaluation are:

- (Primary) Aquifer Parameters
  - The Coefficient of Transmissivity
  - The Coefficient of Storage
- (Secondary) Aquifer Characteristics
  - The Hydraulic Conductivity
  - The saturated thickness of the aquifer

## 3.0 DEFINITIONS

The following definitions are extracted or abstracted from standard references (Section 7); further discussions are available in those references.

Darcian Conditions - Darcian conditions are found where a saturated groundwater system has established an actual or potential flow regime under a head developed within that groundwater system, according to the provisions of Darcy's Law and within the boundary conditions for that law.

Gravity Flow - Gravity flow (vadose conditions) is found where a saturated or unsaturated groundwater system lies within a regime where movement by gravity drainage and capillarity dominate.

Coefficient of Transmissivity (T) - The transmissivity (T) is the rate (for example, in gallons per day per foot of drawdown [gpd/ft]) at which water of the prevailing kinematic viscosity is transmitted through a unit width of the aquifer under a unit hydraulic gradient (Lohman 1979). The transmissivity is mathematically equivalent to the hydraulic conductivity multiplied by the saturated thickness: $T = Kb$.

Coefficient of Storage (S) - The storage coefficient (S) is the (dimensionless) volume of water an aquifer releases from or takes into storage per unit surface area of the aquifer per unit change in head (Lohman 1979).

Hydraulic Conductivity (K) - A medium has a hydraulic conductivity (K) of unit length per unit time (for example, feet per day (ft/d)) if it will transmit in unit time a unit volume of groundwater at the prevailing viscosity through a cross-section of unit area, measured at right angles to the direction of flow, under a hydraulic gradient of unit change in head through unit length of flow (Lohman 1979).

Saturated Thickness (b) - The saturated thickness (b) is the distance (for example, in feet [ft]) from the elevation of the upper groundwater surface in either a phreatic system (the water table) or a confined or semiconfined system (the lower boundary of the upper confining or semiconfining layer, but not the potentiometric surface in a well) to the elevation of the upper boundary of the lower confining or semiconfining layer for the aquifer or water-bearing layer.

Production (Discharge/Injection) Well - The production or discharge/injection well in a pump-test is the well from which water is extracted or into which water is injected for the test.

Observation Well - An observation well is sited near the production well, with an interception interval within, or (at least potentially) in hydrologic or hydraulic communication with, the water-bearing layer intercepted by the production well.

Radial Distance (r) - The radial distance (r) used in pump-test calculations is the horizontal distance, usually in feet (ft), between the production well and the specified observation well.

Drawdown (s) - The drawdown (s) in any well affected by a pump-test is the differential distance, usually in feet (ft), between the static (unstressed) water level in the production or observation well measured immediately prior to the test, and the (stressed) water level at the specified time during the test.

Discharge/Injection Test - The discharge/injection test stage is the part of the pump-test during which water is withdrawn from or injected into the water-bearing layer. The rate of discharge/injection (Q) is typically reported in gallons per minute (gpm) and is usually constant across this part of the pump-test; a stepped test may also be conducted, where Q is varied in a controlled manner.

Recovery Test - The recovery test stage follows cessation of discharge/injection during the pump-test, and, for calculation, uses the Q value of the constant discharge/injection test stage or a weighted value of Q of the stepped test.

Confined Conditions - Confined conditions in a water-bearing layer are found where the groundwater is bounded vertically by opposed surfaces or layers that are impermeable to water, and where the total head of the system at the upper surface of the groundwater is greater than atmospheric pressure. For a confined system, when a well is drilled below the bottom of the upper confining layer, the water level in the well rises to an elevation (at least) within or (possibly) above the upper confining layer.

Unconfined (Phreatic) Conditions - Unconfined conditions in a water-bearing layer are found where the groundwater is bounded vertically only by a single surface or layer at the bottom of the water-bearing layer that is impermeable or semipermeable to water, and where the total head of the system at the upper surface of the groundwater is equal to atmospheric pressure. For an unconfined or phreatic or water-table system, when a well is drilled below the upper surface of the groundwater, the water level in the well does not rise to a significantly higher elevation.

Semiconfined Conditions - Semiconfined conditions in a water-bearing layer are found where the groundwater is bounded vertically by opposed surfaces or layers that are less permeable to water than the water-bearing layer itself, and where the total head of the system is greater than atmospheric pressure. For a semiconfined system, when a well is drilled below the bottom of the upper semiconfining layer, the water level in the well rises to an elevation within or above the upper semiconfining layer. However, one or both of the semiconfining layers will be, in some fashion, in hydraulic and hydrologic communication with the water-bearing layer, and may contribute water to or receive water from that layer.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other, approved procedures are developed. The Project Manager is responsible for development of documentation procedures which deviate from those presented herein.

Site Manager - It is the responsibility of the Site Manager to ensure that the procedures herein are implemented in the field and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures.

Field Geologist - Responsible for determining the need for hydrogeologic testing and has overall responsibility for the planning and implementation of the test. Evaluation and interpretation of the data is also the responsibility of the Field Geologist.

Program Geohydrologist - Responsible for QA/QC oversight of the planning and implementation of the test, along with the evaluation of data generated by the test.

## 5.0 PROCEDURES

The procedures presented in this section concern the administration and execution of pump-tests; the technical content of a given test will be established by the project and program management for each instance according to experience and best professional practice.

### 5.1 Overview

The pump-test will conform to the objectives of the investigation and to standards of good practice common in geohydrologic investigations. Sufficient personnel, and sufficient standard and special equipment will be available for the intentions of the test. Data collection will conform to the practice described in SOP F202 (Water Level, Water/Product Level Measurements and Well Depth Measurements); additionally, time will be measured and recorded no less precisely than the nearest minute or half-minute, as appropriate, and pumpage rates will be measured to the limits of accuracy of the meter while conforming to the intent of the test. Containment and disposal of discharged liquids will conform to the practice described in SOP F504 (Handling of Site Investigation Wastes).

## 5.2 Applications

The pump-test will usually be divided into five stages:

1. Static measurement
2. Preliminary test
3. Equilibration
4. Discharge/injection test
5. Recovery test

The second and third stages may be deleted or expanded.

### 5.2.1 Static Measurement

This stage of the pump-test provides the data on static conditions to be used in subsequent calculation of the aquifer parameters. The static water levels are to be measured no later than immediately prior to the preliminary tests or to the first discharge/injection stage of testing in all wells available for the test. The levels also should have been measured once daily, if possible, for two or more days preceding the test; the optimal measurement program would provide continuous measurement and recording of levels in all wells to be used for a period of several weeks preceding pumpage/injection.

### 5.2.2 Preliminary Test

Preliminary tests will consist of short periods of pumpage or injection at one or a number of rates of discharge/injection. The objectives will be to:

- Predict a probable rate or successive rates of discharge/injection that can maintain a total variation of drawdown or impression within 50 to 75 percent of the available range (the discharge/injection test stage itself can accommodate up to 100 percent of this range);
- Discover observation wells responding especially rapidly or in an exaggerated fashion to the stress induced by discharge/injection; and,
- Provide a general trial of the test system (in particular, that all piping is secure, that all devices perform properly and that the discharge from the pumping well does not provide recharge to the water-bearing layer).

### 5.2.3 Equilibration

The equilibration stage follows the preliminary test to allow return to similar conditions of static levels as those measured before the preliminary test. Calculation of the main test while the water-bearing layer is recovering from the effects of the preliminary test is unnecessarily tedious and subject to error.

### 5.2.4 Discharge/Injection Test

The discharge/injection stage of the pump-test imposes a stress on the water-bearing layer by withdrawing or injecting water at one or more points (the production well or well-field). The measurements of the rates of discharge/injection and of the drawdown in the observation wells provide data used in calculation of the aquifer parameters. The test should be planned to use between 50 and 75 percent of the available drawdown in the production well, but may use up to 100 percent, at the discretion of the Site Manager and Field Geologist.

The rate of discharge/injection may be constant or systematically varied. The duration of this stage of the test will be a marginally acceptable minimum of eight hours, with a preferred minimum of 24 hours for tests under confined conditions and a preferred minimum of 72 hours for tests of unconfined conditions. (This stage can be extended to three or more weeks, according to the intentions of the site investigation.)

### 5.2.5 Recovery Test

The recovery stage will last a minimum of 200 minutes after cessation of discharge/injection, and usually eight hours. This stage monitors the return to equilibrium of the stressed water-bearing layer. Calculations on this stage of the test are used to corroborate or replace the calculations on data from the discharge/injection test; under certain conditions, this stage provides more reliable information than the discharge/injection stage.

## 5.3 Measurements and Measurement Intervals

### 5.3.1 Water Levels

The measurement intervals for water levels in responding observation wells during the discharge/injection stage and in both the production well and the responding observation wells during recovery will be modified from the following suggestions:

| Time Since Start of Discharge/Injection or Recovery (min) | Measurement Frequency (min) |
|---|---|
| 0-5 | 0.5 |
| 5-10 | 1 |
| 10-20 | 2 |
| 20-50 | 5 |
| 50-100 | 10 |
| 100-200 | 20 |
| 200-500 | 50 |
| 500-1000 | 100 |
| 1000-2000 | 200 |
| 2000-5000 | 500 |
| 5000-10000 | 1000 |
| 10000-20000 | 2000 |
| 20000-50000 | 5000 |

Since the crew for the test must monitor conditions in addition to the water levels, a reading interval of not more than 100 minutes is usually reasonable. The actual time and the test time for each reading will be recorded, with the water level measured to a precision of 0.01 ft.

The sequence of stations read and the frequency of readings will be established by project and program management prior to the test, and will be adjusted according to site conditions during the test.

### 5.3.2 Flow Rates

Flow rates for the discharge/injection stage of the test will be measured by a suitable device. Readings will usually be made at intervals of not more than 100 minutes, possibly following an initial period of more frequent readings while the pump stabilizes.

### 5.3.3 Field Parameters

The field parameters of temperature, specific conductance and pH (SOP F201) for the discharge/injection stage of the test will be measured by suitable devices. Readings will usually be made at intervals of not more than 1000 minutes, possibly following an initial period of more frequent readings while the pump stabilizes.

### 5.3.4 Surveying

The radial distance between the production well and an observation well will be measured to a precision of not less than one percent, either by chaining or inspection, or by land survey. The measuring points for water levels need not be surveyed to an accuracy of 0.01 feet, although this is highly desirable for other reasons related to the site investigation.

## 5.4 Calculation Methods

Calculation of the aquifer parameters will follow standard practice, with particular reference to the resources of Section 7, or as otherwise noted in the calculation sequence. A computer program, AQTESOLV (Duffield and Rambaugh) or similar or equivalent program, may also be used, provided that not less than 10 percent of the stations monitored also are calculated by traditional methods.

The calculations will particularly note if the test performance or the resultant calculations indicate a departure from Darcian conditions into vadose flow. Should this be decided, a statement will be made that, although calculations can be made, the calculations are invalid and the values are only roughly approximate; in such case, no valid calculation can be made and no similar pump-tests should be planned for the particular site or area of a site.

## 6.0 QUALITY ASSURANCE RECORDS

The readings made during the pump-test may be recorded in Field Logbooks or on separate forms, according to management decisions. The Field Logbooks will be stored according to SOP F303, with photocopies of the specific pages with pump-test data included in the file for each test. The file for each test will include the field data, the calculations and graphs, and summaries with references for calculations by computer program.

## 7.0 REFERENCES

Chow, V.T. 1964. Handbook of Applied Hydrology. McGraw-Hill, New York.

Lohman, S.W. 1979. Ground-Water Hydraulics. Geological Survey Professional Paper 708. U.S. Government Printing Office.

Freeze, R.A. and Cherry, J.A. 1979. Groundwater. Prentice-Hall, Englewood Cliffs.

Driscoll, F.G., ed. 1986. Groundwater and Wells, 2nd Ed. Johnson Filtration Systems, St. Paul.

Duffield, G.M., Rambaugh, J. O. 1989. AQTESOLV. Aquifer Test Solvent, Version 1.00 Documentation.

# F402
# *SLUG TESTING*

# WELL-HEAD TESTING
# (SLUG-TESTS)

## 1.0 PURPOSE

This SOP provides a general description of the technical methods and field procedures of a representative suite of well-head testing (slug tests) to approximate part of the aquifer parameters. The well-head tests are to be considered at all times as a reconnaissance of the aquifer parameters across an area (the site under investigation); they are never reliable as definitive calculations of those parameters either at a point (an individual well) or across an area (the well-field). Aquifer testing (pump-tests) to calculate these parameters is discussed in SOP F401. The descriptions herein are general in nature and do not apply to a specific well, well-field or project. Prior to designing well-head tests as part of a site investigation and during execution of the tests, the Project Manager, Site Manager and Program Geohydrologist must consult on the appropriate procedures; these procedures must then be recorded in the project documents.

## 2.0 SCOPE

The well-head tests apply both to consolidated and unconsolidated strata; and to confined, semiconfined and phreatic conditions. The aquifer parameters subject to evaluation and approximate calculation are the Coefficient of Transmissivity and the Hydraulic Conductivity.

## 3.0 DEFINITIONS

The following definitions are extracted or abstracted from standard references (Section 7); further discussions are available in those references.

Hydraulic Conductivity (K) - A medium has a hydraulic conductivity (K) of unit length per unit time (for example, feet per day [ft/d]) if it will transmit in unit time a unit volume of groundwater at the prevailing viscosity through a cross-section of unit area, measured at right angles to the direction of flow, under a hydraulic gradient of unit change in head through unit length of flow (Lohman 1979).

Coefficient of Transmissivity (T) - The transmissivity (T) is the rate (for example, in gallons per day per foot of drawdown [gpd/ft]) at which water of the prevailing kinematic viscosity is transmitted through a unit width of the aquifer under a unit hydraulic gradient (Lohman 1979). The transmissivity is mathematically equivalent to the hydraulic conductivity multiplied by the saturated thickness: $T = Kb$.

Saturated Thickness (b) - The saturated thickness (b) is the distance (for example, in feet [ft]) from the elevation of the upper groundwater surface in either a phreatic system (the water table) or a confined or semiconfined system (the lower boundary of the upper confining or semiconfining layer, but not the potentiometric surface in a well) to the elevation of the upper boundary of the lower confining or semiconfining layer for the aquifer or water-bearing layer.

Drawdown (s) - The drawdown (s) in any well affected by a well-head test is the differential distance, usually in feet (ft), between the static (unstressed) water level in the well measured immediately prior to the test, and the (stressed) water level at the specified time during the test.

Falling-Head Test - The falling-head test is conducted where the static water level in the subject well is nearly instantaneously displaced vertically upward at the initiation of the test; the decay of this artificially impressed head is measured against time to provide data for the calculation of conductivity or transmissivity.

Rising-Head Test - The rising-head test is conducted where the static water level in the subject well is nearly instantaneously displaced vertically downward at the initiation of the test; the decay of this artificially depressed head is measured against time to provide data for the calculation of conductivity or transmissivity.

Confined Conditions - Confined conditions in a water-bearing layer are found where the groundwater is bounded vertically by opposed surfaces or layers that are impermeable to water, and where the total head of the system at the upper surface of the groundwater is greater than atmospheric pressure. For a confined system, when a well is drilled below the bottom of the upper confining layer, the water level in the well rises to an elevation (at least) within or (possibly) above the upper confining layer.

Unconfined (Phreatic) Conditions - Unconfined conditions in a water-bearing layer are found where the groundwater is bounded vertically only by a single surface or layer at the bottom of the water-bearing layer that is impermeable or semipermeable to water, and where the total head of the system at the upper surface of the groundwater is equal to atmospheric pressure. For an unconfined or phreatic or water-table system, when a well is drilled below the upper surface of the groundwater, the water level in the well does not rise to a significantly higher elevation.

Semiconfined Conditions - Semiconfined conditions in a water-bearing layer are found where the groundwater is bounded vertically by opposed surfaces or layers that are less permeable to water than the water-bearing layer itself, and where the total head of the system is greater than atmospheric pressure. For a semiconfined system, when a well is drilled below the bottom of the upper semiconfining layer, the water level in the well rises to an elevation within or above the upper semiconfining layer. However, one or both of the semiconfining layers will be, in some fashion, in hydraulic and hydrologic communication with the water-bearing layer, and may contribute water to or receive water from that layer.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other, approved procedures are developed. The Project Manager is responsible for development of documentation procedures which deviate from those presented herein.

Site Manager - It is the responsibility of the Site Manager to ensure that the procedures herein are implemented in the field and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures.

Field Geologist - Responsible for determining the need for hydrogeologic testing and has overall responsibility for the planning and implementation of the test. Evaluation and interpretation of the data is also the responsibility of the Field Geologist.

Program Geologist - Responsible for QA/QC oversight of the planning and implementation of the test, along with the evaluation of data generated by the test.

## 5.0 PROCEDURES

The procedures presented in this section concern the administration and execution of well-head tests; the technical content of a given test will be established by the project and program management for each instance according to experience and best professional practice.

### 5.1 Overview

The well-head test will conform to the objectives of the investigation and to standards of good practice common in geohydrologic investigations. Sufficient personnel, and sufficient standard and special equipment will be available for the intentions of the test. Data collection will conform to the practice described in SOP F202 (Water Level, Water/Product Level Measurements and Well Depth Measurements); additionally, time will be measured and recorded no less precisely than the nearest minute or half-minute, as appropriate, while conforming to the intent of the test. Containment and disposal of discharged liquids will conform to the practice described in SOP F504 (Handling of Site Investigation Wastes).

### 5.2 Applications

The well-head test will usually be divided into three stages:

1. Static measurement
2. Falling-head test
3. Rising-head test

Each stage will normally be run for no more than 30 minutes. The water level in the test well should recover to between 90 and 100 percent of static conditions before beginning the next stage. Should the recovery be less than acceptable after 30 minutes from the start of the first stage, or should other field conditions conspire adversely, the second stage will not be run. Measurements of recovery during the first stage may then be extended to 60 minutes.

#### 5.2.1 Static Measurement

This stage of the well-head test provides the data on static conditions to be used in subsequent approximation of the aquifer parameters. The static water levels are to be measured no later than immediately prior to the first stage of the test, whether falling-head or rising-head. The levels should also have been measured once daily, if possible, for two or more days preceding the test; the optimal measurement program would provide continuous measurement and recording of levels in all wells to be used for a period of several weeks preceding well-head testing.

#### 5.2.2 Falling-Head Test

The falling-head stage of the well-head test is usually conducted before the rising-head. This stage imposes a stress on the water-bearing layer by nearly instantaneously injecting water or introducing a solid slug of impermeable material at one point (the test well). This is usually repeated at a large number of the available wells in the well-field. The measurements of the rate of recovery of the drawdown in the well provides data used in approximation of the aquifer parameters. The test should be planned to use between 50 and 75 percent of the available displacement in the well, but may use between 1 and 100 percent, at the discretion

of the Site Manager. The use of a solid slug is favored by the program. The impressed head developed by this test must rise above the top of the well screen.

### 5.2.3 Rising-Head Test

The rising-head stage of the well-head test imposes a stress on the water-bearing layer by nearly instantaneously extracting water or removing a solid slug of impermeable material at one point (the test well). This is usually repeated at a large number of the available wells in the well-field. The measurements of the rate of recovery of the drawdown in the well provides data used in approximation of the aquifer parameters. The test should be planned to use between 50 and 75 percent of the available displacement in the well, but may use between 1 and 100 percent, at the discretion of the Site Manager. The use of a solid slug is favored by the program.

## 5.3 Measurements and Measurement Intervals

The measurement intervals for water levels in the test well during each stage will be modified from the following suggestions:

| Time Since Start of Test (min) | Measurement Frequency (min) |
|---|---|
| 0-5 | 0.5 |
| 5-10 | 1 |
| 10-20 | 2 |
| 20-60 | 5 |

The actual time and the test time for each reading will be recorded, with the water level measured to a precision of 0.01 ft.

The sequence of stations tested and the frequency of readings will be established by project and program management prior to the tests, and will be adjusted according to site conditions during the tests.

## 5.4 Calculation Methods

Calculation of the approximate values of the aquifer parameters will follow standard practice, with particular reference to the resources of Section 7, or as otherwise noted in the calculation sequence. A computer program, AQTESOLV (Duffield and Rambaugh) or similar or equivalent, may also be used; if the computer program is used, an example that has previously been verified by traditional calculation will be run as part of the data from the subject site.

## 6.0 QUALITY ASSURANCE RECORDS

The readings made during the well-head test may be recorded in Field Logbooks or on separate forms, according to management decisions. The Field Logbooks will be stored according to SOP F303, with photocopies of the specific pages with test data included in the file for each test. The file for each test will include the field data, the calculations and graphs, and summaries with references for calculations by computer program.

## 7.0 REFERENCES

Chow, V.T. 1964. Handbook of Applied Hydrology. McGraw-Hill, New York.

Lohman, S.W. 1979. Ground-Water Hydraulics. Geological Survey Professional Paper 708. U.S. Government Printing Office.

Freeze, R.A. and Cherry, J.A. 1979. Groundwater. Prentice-Hall, Englewood Cliffs.

Driscoll, F.G., ed. 1986. Groundwater and Wells, 2nd ed. Johnson Filtration Systems, St. Paul.

Duffield, G.M., Rambaugh, J. O. 1989. AQTESOLV. Aquifer Test Solvert, Version 1.00 Documentation.

# F501
# *DECONTAMINATION OF DRILLING RIGS AND MONITORING WELL MATERIALS*

# DECONTAMINATION OF DRILLING RIGS AND MONITORING WELL MATERIALS

## 1.0 PURPOSE

The purpose of this SOP is to provide a general reference regarding the proper decontamination of drilling rigs and monitoring well materials used in the performance of field investigations.

## 2.0 SCOPE

This procedure addresses drilling equipment, test pit equipment (i.e. backhoe) and monitoring well material decontamination and should be consulted during the preparation of project-specific plans. This procedure does not pertain to personnel decontamination, or to chemical sampling or field analytical equipment decontamination.

## 3.0 DEFINITIONS

Decontamination - Decontamination is the process of removing or neutralizing contaminants which may have accumulated on field equipment. This process ensures protection of personnel from penetrating substances, reduces or eliminates transfer of contaminants to clean areas, prevents mixing of incompatible substances, and minimizes the likelihood of sample cross-contamination.

## 4.0 RESPONSIBILITIES

Project Manager - It is the responsibility of the Project Manager to ensure that project-specific plans are in accordance with these procedures. Documentation should be developed for areas where project plans deviate from these procedures.

Field Team Leader - It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field. The Field Team Leader is responsible for ensuring the field personnel overseeing decontamination activities, and personnel conducting the activities have been briefed and trained to execute these procedures.

Drilling Inspector (Site Geologist, Rig Geologist etc.) - It is the responsibility of the drilling inspector to ensure that the drilling subcontractor follows these, or other project-specific procedures as directed by the Field Team Leader.

## 5.0 PROCEDURE

The various drilling equipment and materials involved with test boring, test pit excavation, subsurface soil sampling, and monitoring well construction must be properly decontaminated to ensure that chemical analysis results reflect actual concentrations present at sampling locations. These procedures will minimize the potential for cross contamination between sampling locations and the transfer of contamination off site.

## 5.1 Equipment

All drilling equipment involved in field sampling activities shall be decontaminated prior to drilling, excavation, or sampling activities. Such equipment includes drilling rigs, backhoes, augers, downhole tools, well casings, and screens. Split-spoon soil samplers and other similar soil sampling devices shall be decontaminated according to the procedures given in SOP F502, Decontamination of Sampling and Monitoring Equipment.

## 5.2 Decontamination Procedures

Prior to drilling, or leaving the site, large equipment not directly utilized for sampling will be decontaminated by steam-cleaning in a designated area. The decontamination procedure consists of steam-cleaning the equipment, using potable water as the steam source, to remove visible signs of soils or wastes, and allowing the equipment to air dry. If necessary, the equipment may be cleaned with a scrub brush and alconox/liquinox-water solution prior to steam cleaning to remove visible signs of contamination.

The steam cleaning area will be designed to contain decontamination wastes and waste waters, and can be a lined, excavated pit or a bermed concrete pad or asphalt pad. For the latter, a floor-drain must be provided which is connected to a holding sump. A shallow, above-surface tank may be used or a pumping system with discharge to a waste tank may be installed.

At certain sites, due to the type of contaminants or proximity to residences, concerns may exist about air emissions from steam cleaning operations. These concerns can be alleviated by utilizing one or more of the following practices:

- Locate the steam cleaning area on site to minimize potential impacts.
- Enclose steam cleaning operations. For example, augers and drilling rods can be steam cleaned in drums. Tarpaulins also can be placed around the steam cleaning area to control emissions.

Decontamination wastes will be collected and contained unless otherwise directed by the Regulatory Agency. The eventual disposition of these wastes will be determined on a project-specific basis, but may include on-site treatment and/or transport off site to an approved treatment/disposal facility.

## 6.0 QUALITY ASSURANCE RECORDS

Rinsate samples may be collected from steam-cleaned equipment as quality assurance records. The frequency of rinsate samples from either drilling tools or well casings/screens shall be specified in the Sampling and Analysis and Quality Assurance Project Plans for a given project, as appropriate. Documentation in the Field Logbook also shall serve as a quality assurance record of decontamination activities.

## 7.0 REFERENCES

None.

# F502
# *DECONTAMINATION OF SAMPLING AND MONITORING EQUIPMENT*

# DECONTAMINATION OF SAMPLING AND MONITORING EQUIPMENT

## 1.0 PURPOSE

The purpose of this SOP is to provide a general methodology and protocol, and to reference information for the proper decontamination of field chemical sampling and analytical equipment.

## 2.0 SCOPE

This procedure applies to all field sampling equipment including, but not limited to, split-barrel soil samplers (split-spoons), bailers, beakers, trowels, filtering apparatus, and pumps. This procedure should be consulted when decontamination procedures are being developed as part of project-specific plans. Additionally, current USEPA regional procedures and decontamination guidance as well as state guidance should be reviewed.

## 3.0 DEFINITIONS

Decontamination - Decontamination is the process of removing or neutralizing contaminants which may have accumulated on field equipment. This process ensures protection of personnel from penetrating substances, reduces or eliminates transfer of contaminants to clean areas, prevents mixing of incompatible substances, and minimizes the likelihood of sample cross-contamination.

## 4.0 RESPONSIBILITIES

Project Manager - It is the responsibility of the Project Manager to ensure that project-specific plans are in accordance with these procedures. Documentation should be developed for areas where project plans deviate from these procedures.

Field Team Leader - It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field. The Field Team Leader is responsible for ensuring field personnel performing decontamination activities have been briefed and trained to execute these procedures.

Sampling Personnel - It is the responsibility of field sampling personnel to follow these procedures, or to follow documented, project-specific procedures as directed by the Field Team Leader.

## 5.0 PROCEDURES

In order to ensure that chemical analysis results reflect actual concentrations present at sampling locations, sampling equipment must be properly decontaminated prior to the field effort, during the sampling program (i.e., between sampling locations) and at the conclusion of the sampling program. This will minimize the potential for cross-contamination between sampling locations and the transfer of contamination off site.

Preferably, sampling equipment should be dedicated to a given sampling location. If this is not possible, equipment must be decontaminated between sampling locations. Sampling personnel must use disposable gloves and change them between sampling locations.

## 5.1 Sampling Equipment Decontamination Procedures

Soil and sediment sampling equipment including, but not limited to trowels, beakers, dredges, etc., shall be decontaminated using the following USEPA procedures.

USEPA

Prior to use, all sampling equipment should be carefully cleaned using the following procedure:

1. Clean with tap water and laboratory detergent using a brush if necessary to remove particulate matter and surface films.
2. Rinse thoroughly with tap water.
3. Rinse with 10 percent nitric acid rinse
4. Rinse thoroughly with distilled-deionized water and allow to air dry.
5. Rinse with methanol or hexane and allow to air dry.
6. Rinse thoroughly with distilled-deionized water and allow to air dry.
7. Wrap with aluminum foil, if appropriate, to prevent contamination if equipment is going to be stored or transported.

* Portable power augers (such as the Little Beaver®) or large soil boring/drill rigs should be cleaned before boring or drilling operations.
* For badly contaminated equipment, a hot water detergent wash may be needed prior to the rinse procedure.
* If the samples will not be analyzed for metals, then steps 3 and 4 may be omitted; if samples will not be analyzed for organics, then step 5 may be omitted. All solvents must be pesticide-grade.

## 5.2 Field Analytical Equipment Decontamination

Field analytical equipment which may come in direct contact with the sample or sample media, including, but not limited to water level meters, water/product level meters, pH or specific ion probes, specific conductivity probes, thermometers, and/or borehole geophysical probes must be decontaminated before and after use, according to the procedures outlined in Section 5.1, unless manufacturers instructions indicate otherwise. Probes that contact water samples not used for laboratory analyses may be rinsed with distilled water. Probes which make no direct contact (e.g. HNu or OVA probes) will be wiped clean with clean paper towels or an alcohol-saturated cloth.

## 6.0 QUALITY ASSURANCE RECORDS

Decontamination procedures are monitored through the collection of equipment rinsate samples and field blanks. Collection of these samples shall be specified in the project-specific Sampling and Analysis and Quality Assurance Plans. Documentation recorded in the Field Logbook also shall serve as a quality assurance record.

## 7.0 REFERENCES

U. S. EPA Office of Waste Program Enforcement, 1986. RCRA Ground Water Monitoring Technical Enforcement Guidance Document (TEGD). OSWER Directive 9950.1.

U. S. EPA, 1991. Standard Operating Procedures and Quality Assurance Manual. Environmental Compliance Branch, U. S. EPA Environmental Services Division, Athens, Georgia.

Micham, J. T., R. Bellandi, E. C. Tifft, Jr., Spring 1989. "Equipment Decontamination Procedures for Ground Water and Vadose Zone Monitoring Programs: Status and Prospects." in Ground Water Monitoring Review.

# F504
# *HANDLING OF SITE INVESTIGATION WASTES*

# HANDLING OF SITE INVESTIGATION DERIVED WASTES

## 1.0 PURPOSE

The purpose of this SOP is to provide guidance for the disposal of investigation derived wastes (IDW) generated under a field investigation program.

## 2.0 SCOPE

This procedure describes the steps necessary to dispose of site investigation derived wastes that are generated during field investigations. These wastes may be either hazardous or nonhazardous in nature. The nature of the waste (hazardous or nonhazardous) will determine how the wastes will be handled during the field investigation. The sources of waste material depend on the site activities planned for a project. The following types of activities (or sources) that are typical of site investigations, may result in the generation of waste material which must be properly handled:

- Soil borings and monitoring well construction (drill cuttings)
- Mud rotary drilling ( potentially contaminated mud)
- Monitoring well development (development water)
- Groundwater sampling (purge water)
- Heavy equipment decontamination (decontamination fluids)
- Sampling equipment decontamination (decontamination fluids)
- Personal protective equipment [PPE] (health and safety disposables)

## 3.0 DEFINITIONS

Investigation Derived Waste (IDW) - A waste (hazardous or nonhazardous) generated during a field investigative task that has been properly labeled, stored, and containerized while awaiting final disposition. These wastes may include drilling muds, soil cuttings, and purge water from test pit and well installation; purge water, soil, and other materials from collection of samples; residues (e.g., ash, spent-carbon, well development purge water) from testing of treatment technologies and pump and treat systems; contaminated PPE; and solutions used to decontaminate non-disposable PPE and equipment (USEPA, April 1992, Guide to Management of Investigation-Derived Wastes).

## 4.0 RESPONSIBILITIES

Client - The Client must ultimately be responsible for the final disposition of site wastes. As such, a facility representative will usually prepare and sign waste disposal manifests as the generator of the material, in the event off-site disposal is required. However, it may be the responsibility of Baker, depending on the contingency discussions during execution of the investigation to provide assistance to the Client in arranging for final disposition and preparing the manifests.

Project Manager - It is the responsibility of the Project Manager to select investigation methods that minimize the generation of waste material, where possible, and to work with the Client in determining the final disposition of site investigation wastes. The Project Manager will relay the results and implications of the chemical analysis of the waste or associated material, and advise on the regulatory requirements and prudent measures appropriate to the disposition of the material. The Project Manager also is responsible for ensuring that the site manager and/or field team leader for the site, is familiar with the procedures to be implemented in the field, and that all required field documentation has been completed.

Site Manager/Field Team Leader - The Site Manager or Field Team Leader is responsible for the on site supervision of the waste handling procedures during the site investigations. The Site Manager or Field Team Leader also is responsible for ensuring that all other field personnel are familiar with these procedures.

## 5.0 PROCEDURES

### 5.1 Preliminary Activities

Prior to the initiation of site activities the expected sources, media, and method(s) of containerizing and staging of these materials will be identified.

### 5.2 Designation of Potentially Hazardous and Nonhazardous IDW

Wastes generated during the field investigation can be categorized as either potentially hazardous or nonhazardous in nature. The designation of such wastes will determine how the wastes will be handled. The criteria for determining the nature of the waste, and the subsequent handling, is described below for each type of investigative waste.

#### 5.2.1 Drill Cuttings/Mud

Drill cuttings and mud generated during the augering of test (soil) borings and monitoring well installation boreholes, will be containerized in 55-gallon drums or in lined roll-off boxes. As the borehole is augered, and soil samples collected, the site geologist will monitor the cuttings/samples with an HNu photoionization (PID) unit for organic vapors. In addition, the site geologist will describe the soils in a Field Logbook. Upon completion, the soil borings will be backfilled with a cement-bentonite grout.

#### 5.2.2 Monitoring Well Development and Purge Water

All site development and purge water shall be containerized in 55-gallon drums, tankers, or large (250-gallon) containers. 55-Gallon drums will initially be strategically located at the site (i.e., next to each well).

#### 5.2.3 Decontamination Fluids

Equipment and personal decontamination fluids shall be containerized in 55-gallon drums or tanks, if appropriate. The fluids shall be collected from each of the "decon"/wash pads on a daily basis. Decontamination fluids containing solvents and/or acids may be containerized separately.

### 5.2.4 Personal Protective Equipment

All personal protective equipment (e.g., tyvek, gloves, and other health and safety disposables) shall be double bagged and placed in a 55-gallon drum or a dump box, which will be either arranged by Baker or provided by the Client. The Client assisted by Baker will ultimately dispose of these materials.

## 5.3 Containerization

Waste materials should be segregated to minimize disposal quantities of hazardous materials. For instance, soils from a particular boring will be placed in a single set of containers for that boring. Development and purge water from a given well may be placed in the same set of containers; however, water from different wells should be placed in different containers unless otherwise stipulated by the Project Plans.

Polyethylene or other suitably compatible liners will be used in roll-off boxes for solids. The containers are to remain closed except when filling, emptying or sampling. The container lid shall be securely attached at the end of each work day or when the container is completely empty.

## 5.4 Labeling

When 55-gallon drums are used to containerize IDWs, the containers will be closed, numbered and labeled by the field team during the site investigation. Information shall be recorded both on the container lid and its side. Container labels shall include, as a minimum:

- Date
- Site number
- Project number
- Boring or well number
- Matrix (liquid, solid)
- Contents (dev. water, decon fluids, etc.)
- Contaminant of concern (PCBs, solvents, metals, etc.)

If laboratory analysis reveals that containerized materials are hazardous or contain PCBs, additional labeling of containers may be required. The project management will assist the Client in additional labeling procedures, if necessary, after departure of the field team from the facility. These additional labeling procedures will be based upon the identification of material present; EPA regulations applicable to labeling hazardous and PCB wastes are contained in 40 CFR Parts 261, 262 and 761.

## 5.5 Container Storage

Containers of site investigation wastes shall be stored in a designated and secure area that is managed by the client until disposition is determined.

If the laboratory analysis reveals that the containers hold hazardous or PCB waste, additional storage and/or security measures may be implemented; in the absence of the investigation team, this will be the responsibility of Client assisted by Baker.

Baker will assist the Client in devising the storage requirements, which may include the drums being staged for easy access or on wooden pallets or other structures to prevent contact with the ground. Weekly inspections of the temporary storage area by facility personnel may also be required. These inspections may assess the structural integrity of the containers and proper container labeling. Also, precipitation that may accumulate in the storage area may need to be removed. These weekly inspections and precipitation removal events, shall be recorded in the site logbook.

### 5.6 Container Disposition

The disposition of containers of site investigation generated wastes shall be determined by the Client and regulatory personnel with the assistance of Baker, as necessary. Disposition of the containerized waste shall be based on quantity, types of material, and analytical results. If necessary, samples of the containerized waste may be collected for waste characterization purposes. Disposition will not be addressed until after receipt of applicable analytical results; these results are usually not available until long after completion of the field investigation at the facility.

### 5.7 Disposal of Contaminated Materials

Actual disposal methods for contaminated materials disturbed during a site investigation are the same as for other PCB or hazardous substances: incineration, landfilling, treatment, and so forth. The responsibility for disposal must be determined and agreed upon by all involved parties during negotiations addressing this contingency.

The usual course will be a contractor specialist retained to conduct the disposal. However, regardless of the mechanism used, all applicable Federal, state and local regulations shall be observed. EPA regulations applicable to generating, storing and transporting PCB or hazardous wastes are contained in 40 CFR Parts 262, 263 and 761.

Another consideration in selecting the method of disposal of contaminated materials is whether the disposal can be incorporated into subsequent site cleanup activities. For example, if construction of a suitable on-site disposal or treatment structure is expected, contaminated materials generated during the site investigation may be stored at the site for treatment/disposal with other site materials. In this case, the initial containment (i.e., drums or other containers) shall be evaluated for use as long-term storage. Also, other site conditions, such as drainage control, security and soil types must be considered in order to provide proper storage.

## 6.0 QUALITY ASSURANCE RECORDS

A container log shall be maintained in the Site Logbook. The container log shall contain the same information as the container label plus any additional remarks or information. Such additional information may include the identification number of a representative laboratory sample. Weekly inspections of the drum or dump box storage areas will be performed and documented in the site log.

## 7.0 REFERENCES

40 CFR Parts 261, 262, 263 and 761.

# ATTACHMENT A

# GENERALIZED
# SOIL GAS SURVEY STANDARD OPERATING PROCEDURE

# SOIL GAS SAMPLING PROCEDURE

I. Probe Placement

A. A clean probe (pipe) is removed from the "clean" storage tube.
B. The soil gas probe is placed in the jaws of hydraulic pusher/puller mechanism.
C. A sampling point is put on the bottom of the probe.
D. The hydraulic pushing mechanism is used to push the probe into the ground.
E. If the pusher mechanism will not push the probe into the ground a sufficient depth for sampling, the hydraulic hammer is used to pound the probe into the ground. Please note that concrete cutting may be required prior to probe placement pending sampling point location.
F. The hydraulic pushing mechanism is used to pull the probe up a few inches releasing the sampling point.

II. Sample Extraction

A. An adaptor is put onto the top of the soil gas probe.
B. The vacuum pump is hooked onto the adaptor.
C. The vacuum pump is turned on and used to evacuate soil gas.
D. Evacuation will be at least 30 seconds but never more than 5 minutes for samples having evacuation pressures less than 15 inches of mercury. Evacuation times will be at least 1 minute, but no more than 5 minutes for probes reading greater than 15 inches of mercury.
E. Gauges on the vacuum pump are checked for inches of mercury.
    1. Gauge must read at least 2 inches of mercury less than maximum vacuum to be extracting sufficient soil gas to collect a valid sample.

III. Sample Collection

A. With vacuum pump running, a hypodermic syringe needle is inserted through the silicone rubber and down into the metal tubing or adaptor.
B. Gas samples should only contact metal surfaces and never contact potentially sorbing materials (i.e., tubing, hose, pump diaphragm).
C. The syringe is purged with soil gas then, without removing syringe needle from adaptor, a 10 mL soil gas sample is collected.
D. The syringe and needle are removed from the adaptor and the end of the needle is capped.
E. If necessary, a second 10 mL sample is collected using the same procedure.

IV. Deactivation of Sampling Apparatus

A. The vacuum pump is turned off and unhooked from the adaptor.
B. The adaptor is removed and stored with equipment to be cleaned.
C. Using the hydraulic puller mechanism, the probe is removed from the ground.
D. The probe is stored in the "dirty" probe tube.
E. The probe hole is backfilled, if required.

V. Log Book Notations for Sampling

A. Time (military notation)
B. Sample number
C. Location (approximate description – i.e., street names)
D. Sampling depth
E. Evacuation time before sampling
F. Inches of mercury on vacuum pump gauge
G. Probe and adaptor numbers
H. Number of sampling points used
I. Observations (i.e., ground conditions, concrete, asphalt, soil appearance, surface water, odors, vegetation, etc.)
J. Backfill procedure and materials, if used

VI. Other Record keeping

A. Data sheets are filled out, if required
B. Sample location is marked on the site map

Note:

Typical soil gas/in situ groundwater sampling investigations include a Report of Results from the Soil Gas Survey firm. Report format and content may vary; however, project specific requirements (i.e., Record keeping/documentation) should be identified and confirmed prior to on site operations.

# F600
# *SOIL GAS AND IN SITU GROUNDWATER SAMPLING SURVEYS*

# SOIL GAS AND IN SITU GROUNDWATER SAMPLING SURVEYS

## 1.0 PURPOSE

The purpose of this SOP is to describe the methods and procedures involved in conducting a soil gas survey.

## 2.0 SCOPE

The methods described in this SOP are applicable to the detection of groundwater and soil contamination, determining the extent of the contamination, and identifying the source(s) of contamination. Soil gas monitoring provides a quick means of waste site evaluation. Using this method, underground contamination can be identified, and the source, extent, and movement of the pollutants can be traced.

## 3.0 DEFINITIONS

Dynamic Device - A method whereby soil gas samples are extracted through a hollow steel tube using a vacuum extraction pump.

Passive Device - A method where adsorption devices are implanted in the shallow surface soils. These are left in place for days or weeks. After a set time, the devices are dug out and sent out for analysis.

Direct Connection - The direct transfer of soil gas into the on-site gas chromatograph (GC) via a vacuum pump.

Syringe - Device used to transfer the soil gas sample into the GC.

Gas Bulbs/Bags - Devices to hold soil gas samples prior to analysis.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or other approval methods are developed. The Project Manager is responsible for development of documentation of procedures which deviate from those presented herein.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the specific sampling techniques and equipment to be used, and documenting these in accordance with the Sampling and Analysis Plan. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures.

Sampling Personnel - It is the responsibility of the field sampling personnel to follow these procedures, or to follow documented, project-specific procedures as directed by the Field Team Leader and/or the Project Manager. The sampling personnel are responsible for the proper acquisition of soil gas samples.

## 5.0 PROCEDURES

Prior to selecting sample locations, an underground utility search is recommended. The local utility companies or facility utility personnel can be contacted and requested to mark the locations of their underground lines. Sampling plans/designs can then be drawn up accordingly.

Typical methodology involves the insertion of (3/8-inch diameter) probes into the ground to a desired depth via a hydraulic pusher/puller mechanism. A 1/4-inch O.D. stainless steel probe is inserted into the hole and sealed at the top around the probe. The gas contained in the interstitial spaces of the soil is sampled by pulling the sample through the probe using an air sampling pump.

Attachment A is a generalized SOP including quality control procedures performed by soil gas survey firms. The methods and procedures described herein are typical for this type of subsurface investigation.

Some soil gas firms have the ability to collect and analyze groundwater samples using a probe apparatus in a manner similar to collecting soil gas samples. Specific requirements are documented in the Project Plans.

## 6.0 QUALITY ASSURANCE RECORDS

Quality assurance records will vary between soil gas survey firms. Ordinarily QA/QC records accompany soil gas survey firm reports in the form of spike or blank results from analysis of "Field QA Samples" prepared and analysis, as required under each individual project. Project Plans should include QA/QC specific requirements for the soil gas or in situ groundwater sampling program.

## 7.0 REFERENCES

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2. La Fond, J. W. "EmFlux Soil Gas Survey." Quadrel Services, Inc., Jamsville, Maryland. July 30, 1991.

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# F700
# *INTRODUCTION TO GEOPHYSICAL SURVEYS*

# APPLICATION OF GEOPHYSICAL METHODS FOR GROUNDWATER AND HAZARDOUS WASTE SITE ASSESSMENTS

## 1.0 INTRODUCTION

This section of the Standard Operating Procedures (SOP) discusses geophysical exploration methods and their application to characterizing site conditions and evaluating contamination problems. The purpose is to familiarize the reader in general terms with the usefulness of geophysical methods and how data are acquired, processed and interpreted.

A geophysical survey measures the physical properties of earth materials in detail or averaged over relatively large areas. Variations in the electrical field (applied and ambient), gravity and magnetic potentials and seismic wave velocities, amplitudes and frequencies are measured systematically. These properties are affected by the structure and composition of the subsurface soil, rocks, and water. Discontinuities in physical properties often correspond to geological or manmade boundaries. The primary objectives in the use of geophysical methods are to locate and quantify geological and/or manmade features. Depending on "sampling" frequency, geophysical methods measure the detailed or averaged physical properties of materials for the points of observation. All methods are inherently subject to greater averaging and lower resolution as distances between sampling points increase.

Geophysical surveys utilize both active and passive techniques. In an active method, some form of energy is introduced into the subsurface and the response of subsurface materials to energization is measured. Active measurements usually provide the greatest resolution and accuracy. Passive measurements simply record the strengths of natural geophysical fields or changes in field strength.

A geophysical investigation provides both reconnaissance and detailed measurement of critical factors for site assessment in a rapid, cost effective manner to allow an evaluation of subsurface conditions. The results of a geophysical investigation are used to:

- Determine the source and extent of contamination problems
- Characterize geologic conditions
- Optimize test pit and boring locations

In most cases the proper application of a geophysical investigation adds significant information and reduces the costs necessary to acquire the information required to determine an effective site remediation and cleanup. The correlation of data from two or more independent methods, plus borehole geologic and sampling data, provides the most meaningful results.

Results of a geophysical site investigation usually cannot provide a complete analysis of a groundwater related problem. A drilling program usually is necessary to supplement a geophysical program. Results of the geophysical program can minimize the number of borings necessary and optimize their locations. In return, the borings provide important data for correlation with geophysical results. In some instances, geophysical methods measure average subsurface conditions for a large area, while borings provide detailed information for a limited area. A combined geophysical-boring program is a cost effective system for the most complete analysis of site conditions. Often the geophysical survey provides significant cost savings in providing information that allows borings and test pits to be located where they will do the most good.

In order to effectively plan a geophysical site investigation, the following topics must be addressed:

- Purpose of the Investigation
- Geologic Conditions
- General Site Conditions
  (topography, nearby metal objects, power lines, buried utilities, etc.)
- Site History

This information is needed to select the most effective geophysical methods for the investigation and to estimate the extent of the coverage necessary. Geophysical methods are selected by their sensitivity to the problem and by their insensitivity to site noise (interference) conditions. Generally, multi-technique investigations are desirable. The commonly used geophysical methods and their advantages and limitations are described in Table I.

Texts that generally discuss the applicable geophysical techniques include Dobrin (1976), Telford and others (1976), Mooney (1977), U.S. Army Corps of Engineers (1979), Grant and West (1965), and Griffiths and King (1981). A comprehensive discussion of geophysical methods and their application to ground water problems is included in the (1985) Electric Power Research Institute's Ground Water Investigation and Mitigation Techniques, Section 3. Additional sources of information for specific methods are referenced in the discussions of each geophysical method.

TABLE I

COMPARISON OF GEOPHYSICAL METHODS FOR HAZARDOUS WASTE APPLICATION

| Geophysical Method | Cost | General Applications | Relative Depth of Investigation | Advantages | Disadvantages | Accuracy |
|---|---|---|---|---|---|---|
| Seismic Refraction | Moderate | Determine depths to bedrock and water table. Identify zones of fractured and/or weathered bedrock. | 0 to 10 meters | Accurately identifies groundwater and rock layering. | Sensitive vibration, construction actlvities and electrical noise. | ±10% of depth to interface |
| Electrical Resistivity | Inexpensive | Determine depths to water table, clays, and bedrock. | 10 to 100+ meters | Easy to operate. Equipment is inexpensive. | Dipping strata complicates interpretatlon. | ±25% of total depth |
| Self Potential | Inexpensive | Identify groundwater flow and area of contamination. | 0 to 20 meters | Equipment is inexpensive. Easy to operate. Highly qualitative interpretation. | Susceptible to interference from lithological and vegetation differences. | |
| Electromagnetic | Moderate | Plume detection and tracing. Depths to water table, bedrock, clays, etc. | 0 to 5 meters | Walk over method. | Lacks the resolution and depth. | ±25% accuracy |
| Ground Penetrating Radar (GPR) | Moderate | Buried metals detection and general identification. | 0 to 10 meters | Tow-along method. Radar commercially available. Easy to operate and high resolution. | Depth of penetration limited by conductivity of material (drum, tank, debris, etc.). | ±20% accuracy |
| Magnetics Equipment | Inexpensive | Buried ferrous detection. | 0 to 10 meters | Easy to operate and equipment commercially available. | Sensitive to fences, power lines, pipes, and cultural metal ferrous objects. | |
| Gravity | High | Detection of joints, fault scapes, buried river channels, collapse or fill areas. | 10 to 100 meters | Data can be acquired in highly developed urban areas. Field work done by one person. Equipment is portable. | Instrument and survey equipment expensive. Geologic data necessary for interpretation. Also, requires highly trained field personnel. | |
| Borehole Measurement | Moderate | Drill to known depth of measurement. | 5 to 100+ meters | Very good vertical resolution. Most equipment easy to operate. Little data reduction. | Borehole construction may limit techniques. Radioactive tools need special licensing. | |

Note: All methods can be operated in a non-intrusive manner.

# F701
# *GEOPHYSICS: BOREHOLE*

# GEOPHYSICS: BOREHOLE

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information for using borehole geophysical logging.

## 2.0 SCOPE

This SOP indicates the normal suite of logging that may be used, as well as certain supplemental logs. The description of logging techniques includes the type of device, the physical property responding to or recorded by the device, and the general pattern of interpretation and correlation related to the device or the suite of logs.

## 3.0 DEFINITIONS

Active technique - A technique in which a stress is applied to the material under study and the resultant response is measured. Stresses can include electrical current, sound waves or neutron or gamma ray bombardment.

Calibration - The process of checking and adjusting the tool reading to a standard of known value.

Lithology - The physical character of a rock or rock type; also often used for describing overburden material (e.g., sand, clay, till).

Measuring point - The point, on a probe or sonde device, where the reading is taken (e.g., the tips of the caliper arms, the detector on a gamma-ray tool).

Non-unique response - Tool response that is not unique to a specific rock characteristic. For example, several different rock types exhibit low gamma-ray counts; water-filled fractures and clay layers both have low resistivity values.

Passive technique - A technique which measures properties inherent to the material; examples include self-potential, gamma-ray, temperature.

Probe - The downhole electronics and detecting/measuring apparatus of the logging system, usually encased in a stainless steel jacket.

Reference elevation - The surface elevation which acts as a common measuring point for all correlations, commonly ground surface or top of casing.

Resolution (vertical) - Ability to see thin layers.

Sonde - same as probe.

Total depth - The deepest point in the boring as determined by geophysical logs.

## 4.0 RESPONSIBILITIES

Project Manager – The Project Manager is responsible for ensuring that the project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for ensuring that the personnel operating and interpreting the geophysical data are trained, skilled in that endeavor, so far as to receiving documentation on the training and experience of the operating personnel.

Field Team Leader – The Field Team Leader is responsible for selecting and detailing the borehole technique and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the borehole logging activities have been briefed and trained to execute these procedures.

## 5.0 PROCEDURES

### 5.1 Overview

Borehole geophysical techniques (also called logging) are a group of active and passive geophysical methods used to provide detailed measurements of soil, rock and water properties. This is done by lowering tools into the borehole to measure the electrical, acoustical or radioactive properties of the materials surrounding a borehole. The surveys are non-destructive and can often be run in existing boreholes and water supply wells with no modifications.

The techniques are based on counterpart surface geophysical methods, adapted to the borehole environment (Table 1). Typically, these adaptations include the reduction of equipment size (most techniques will fit inside a two-inch diameter hole), reduction and standardization of the fixed source to receiver spacing (and a corresponding reduction in how deep the technique looks into the formation), protection of tools from pressure and temperature effects, and interpretation of data with respect to vertical rather than horizontal changes.

The relation of borehole geophysical techniques as investigative suites, and the responses interpreted, are indicated on Table 2 for the usual and some unusual applications.

### 5.2 Borehole Applications

#### 5.2.1 Self-Potential

- Identification of zones of water loss or gain (streaming potential)
- Qualitative indication of clay content/determination of clay layers
- Qualitative indication of water salinity
- Rock type correlation/layer thickness

#### 5.2.2 High-Resolution Caliper

- Used in open borehole geophysics
- Provides rock density in grams per cubic centimeter
- Provides borehole diameter

### 5.2.3 Natural Gamma

- Yields a measure of potassium-40 content in rocks
- High clay content
- Permeability

### 5.2.4 Resistivity Profiling

- Provides a measure of clay content in geologic materials
- Can be used as an olimeter

### 5.2.5 Temperature Log

- Yields temperature at a specific vertical location in a borehole to presence of groundwater
- Measures inflow and outflows of a borehole/well

### 5.2.6 Fluid Conductivity Meter

- Yields direct iron content of groundwater by measuring conductivity of the fluid in which the probe is immersed

### 5.2.7 Neutron Tool

- Measures porosity of geologic materials by sending neutrons into the materials. Higher porosity will be indicated by return of fewer neutrons to the tool.

### 5.2.8 Long-Space Density

- Measures vertical flow in a borehole/well

### 5.2.9 Flow Meter

- Measures flow across a borehole/well

## 6.0 QUALITY ASSURANCE RECORDS

Data will be recorded in log books or on data log sheets attached to the monitoring device. All data will be entered with the following: date, location, personnel on site, start and end time (in military time) and weather.

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# F702
# *ELECTROMAGNETIC INDUCTION METHOD*

# GEOPHYSICS: ELECTROMAGNETIC INDUCTION METHOD

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information for using electromagnetic induction (EM) methods.

## 2.0 SCOPE

This SOP provides a description of field procedures, equipment, and interpretation methods necessary to fully utilize this procedure.

## 3.0 DEFINITIONS

Conductivity – Ability of a material to transmit an electrical current. Inverse of resistivity.

Horizontal dipole mode – Transmitter and receiver coils oriented vertically.

Vertical dipole mode – Transmitter and receiver coils oriented horizontally.

Vertical sounding – Multiple EM measurements centered at a point with varying coil spacings.

Vertical profiling – EM measurements along a traverse with a fixed coil spacing and coil orientation.

## 4.0 RESPONSIBILITIES

Project Manager – The Project Manager is responsible for ensuring that the project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for ensuring that the personnel operating and interpreting the geophysical data are trained, skilled in that endeavor, so far as to receiving documentation on the training and experience of the operating personnel.

Field Team Leader – The Field Team Leader is responsible for selecting and detailing the geophysical technique and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the activities have been briefed and trained to execute these procedures.

Field Investigation Personnel – It is the responsibility of the field investigation personnel to follow these procedures, or to follow documented, project-specific procedures as directed by the Field Team Leader and the Project Manager. Field personnel are responsible for the proper acquisition of geophysical data.

## 5.0 PROCEDURES

### 5.1 Overview

Electromagnetic Induction (EM) methods are non-intrusive geophysical techniques of measuring the apparent conductivity of the subsurface materials. Electrical conductivity values of subsurface materials are determined by transmitting a high frequency electromagnetic (primary) field into the earth and measuring the secondary electromagnetic field produced by the eddy current as illustrated in Figure 1. The transmitter

and receiver coils do not require direct ground contact thus permitting continuous profiling and rapid data acquisition.

$$resistivity\ (ohm\text{-}meters) = \frac{1{,}000}{EM\ instrument\ readout\ (milliohms\ per\ meter)}$$

The strength of the secondary field is a function of the inter coil spacing, operating frequency and ground conductivity. The ratio of the secondary to the primary magnetic field is directly proportional to the terrain conductivity which enables direct instrument readout of apparent conductivity values (measured conductivity values are the bulk average conductivity for the area or volume of earth sampled). Conductivity ranges typical of various earth materials are shown on Figure 2. EM conductivity values are usually expressed in units of milliohms per meter. Conductivity values are converted to resistivity values in ohm-meters by use of the following relationship: The apparent conductivity of the subsurface materials is dependent upon subsurface conditions such as:

- Lithology
- Porosity
- Permeability
- Conductivity of subsurface pore fluids

Changes in these parameters causing measurable variations in electromagnetic conductivity can result from:

- Conductive contaminant plumes
- Abandoned trenches and lagoons
- Lateral changes such as backfill or landfill materials
- Bedrock fracture zones
- Lithological variations
- Buried metallic objects

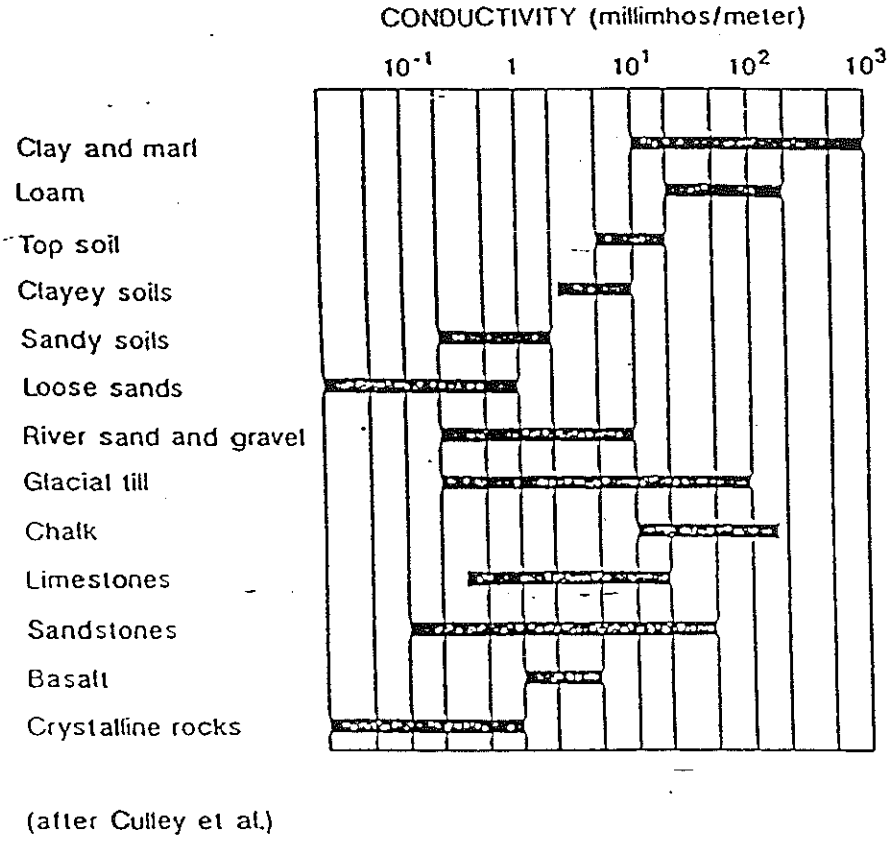
The sampling depth or depth of investigation is related to the coil spacing and coil mode. The two coil modes used are the vertical dipole mode (coils horizontal) and the horizontal dipole mode (coils vertical). Figure 3 shows the relationship of the coil spacings, mode and relative responses.

Two common terrain conductivity meter are EM-31 and the EM-34-3. The EM-31 has a fixed intercoil spacing of 3.7 meters and an effective depth of penetration of approximately 6 meters. The EM-34-3 has two coils which can be separated by 10, 20, or 40 meters and can be oriented in either the horizontal or vertical dipole modes. Intercoil separations increase the effective depth of investigation as shown below.

| Intercoil Spacing | Depth of Investigation (meters) | |
|---|---|---|
| (meters) | Horizontal Dipole | Vertical Dipoles |
| 10 | 7.5 | 15 |
| 20 | 15 | 30 |
| 40 | 30 | 60 |

The coil orientation (horizontal or vertical) allows the EM-34-3 to respond to materials of different depths.

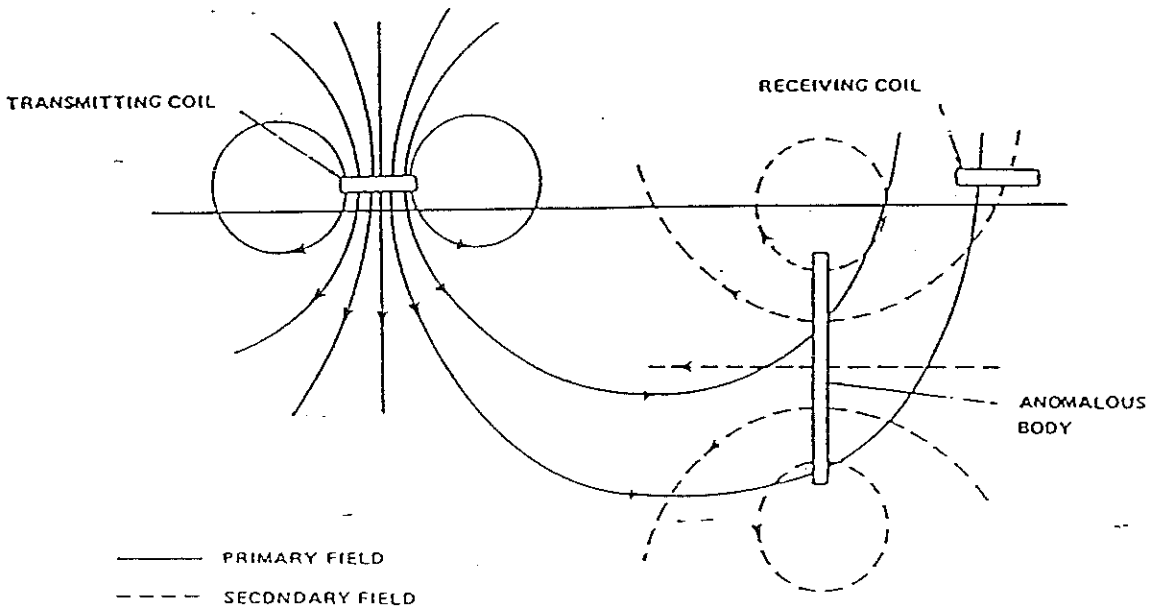
Figure 1




(after Culley et al.)

CONDUCTIVITY RANGES FOR COMMON EARTH MATERIALS

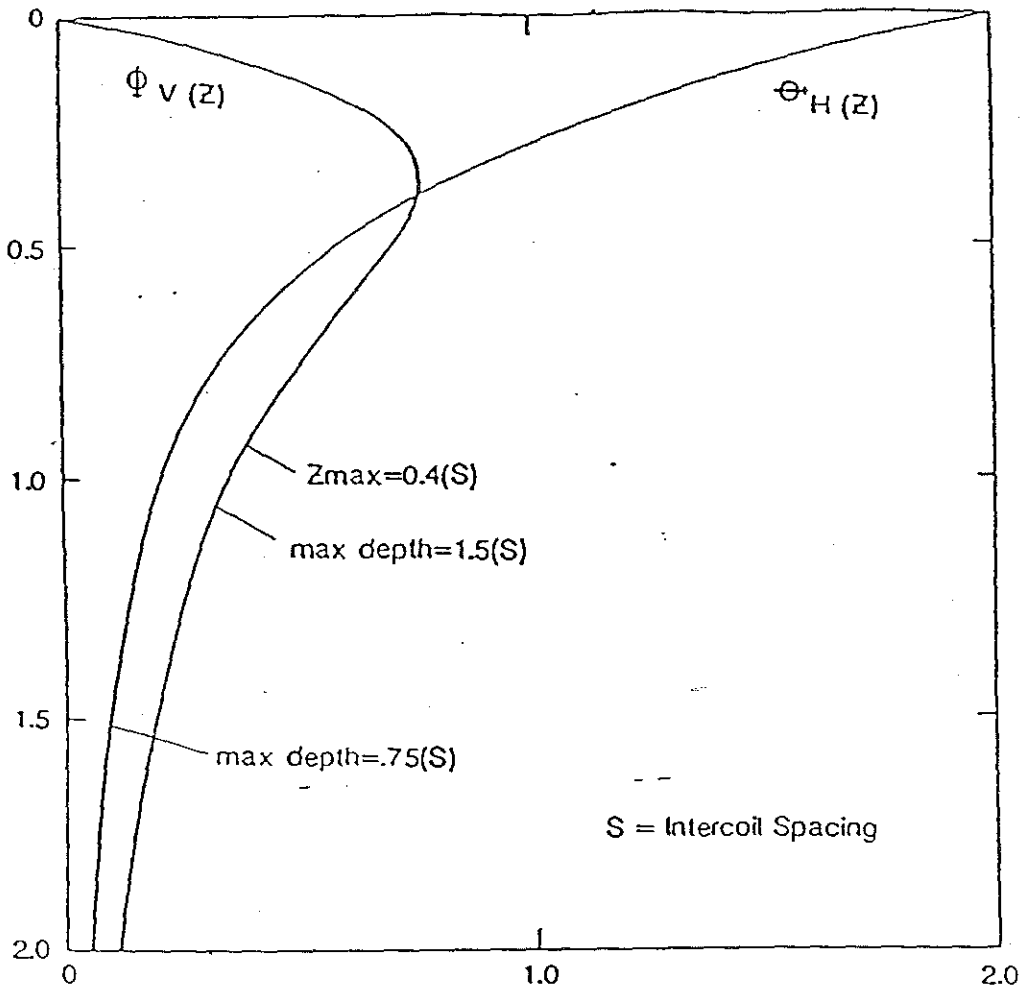
Figure 2




Source: Griffith and King, 1981

TWO-COIL ELECTROMAGNETIC INDUCTION APPARATUS

Figure 3




COMPARISON OF RELATIVE RESPONSES FOR VERTICAL AND HORIZONTAL DIPOLES

Vertical sounding and horizontal profiling are the two EM survey techniques. Vertical profiling is accomplished by multiple measurements about a point with varying coil spacing. Horizontal profiling is performed by making measurements along traverses with a fixed coil spacing. General discussions of electromagnetic induction methods are presented in texts by Grant and West (1965), Telford and others (1976), and Griffiths and King (1981).

## 5.2 Applications and Uses

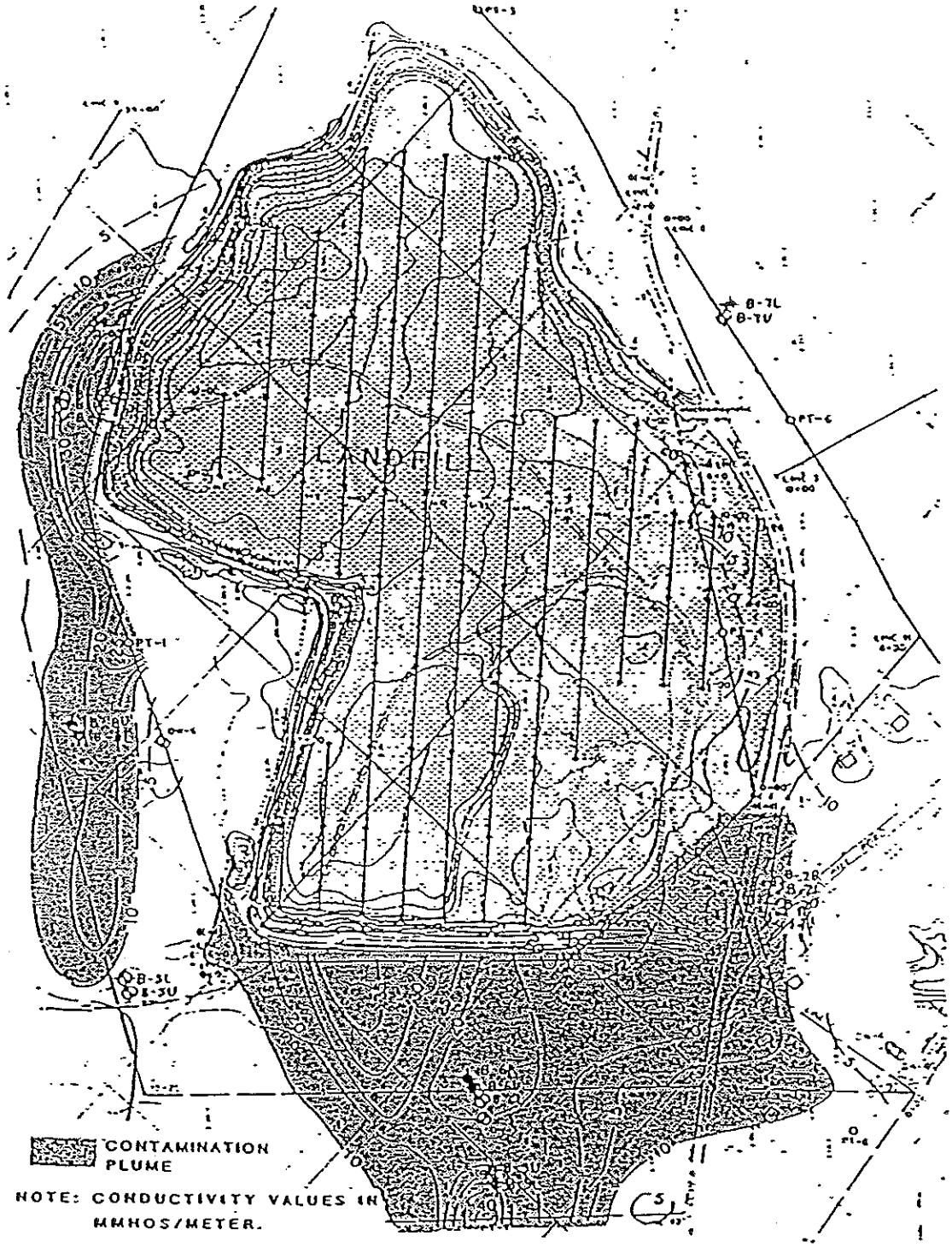
The measurement of subsurface conductivity at a hazardous waste site provides a valuable contribution to site characterization. The conductivity (resistivity) of the hydrogeologic section is predominantly influenced by the pore fluids. Consequently, conductivity measurements provide indirect information on the porosity and permeability of subsurface materials, the degree of saturation, and the conductivity of the pore fluids. The conductivity of the pore fluid is influenced by the presence of dissolved electrolytes. Contaminant plumes in the unsaturated and saturated zones can be mapped provided there is a sufficient change in the conductivity to be detected by the EM instrument. Generally, contaminant plumes of inorganic waste are easily detected because the pore fluids often have conductivity values as much as three orders of magnitude above background values. Figure 4 illustrates an EM anomaly associated contamination plume. EM conductivity measurements can also be used to detect the presence of buried waste; filled disposal trenches, and buried metal objects such as drums, tanks or metal debris. Figure 5 illustrates an EM anomaly over a buried metal object. Electromagnetic surveys can be used to locate conductive as well as and non-conductive bodies. The many applications include:

- Contaminant plume mapping
- Locating abandoned trenches and lagoons
- Delineating bedrock fracture zones
- Determining thickness of weathered layers
- Lithology mapping
- Locating buried metallic objects
- Lateral anomalies such as pockets or pits of different materials

Examples of EM applications at sites where groundwater is contaminated are presented by Duran (1982), Greenhouse (1983), and Greenhouse and Slaine (1983).

Figure 4

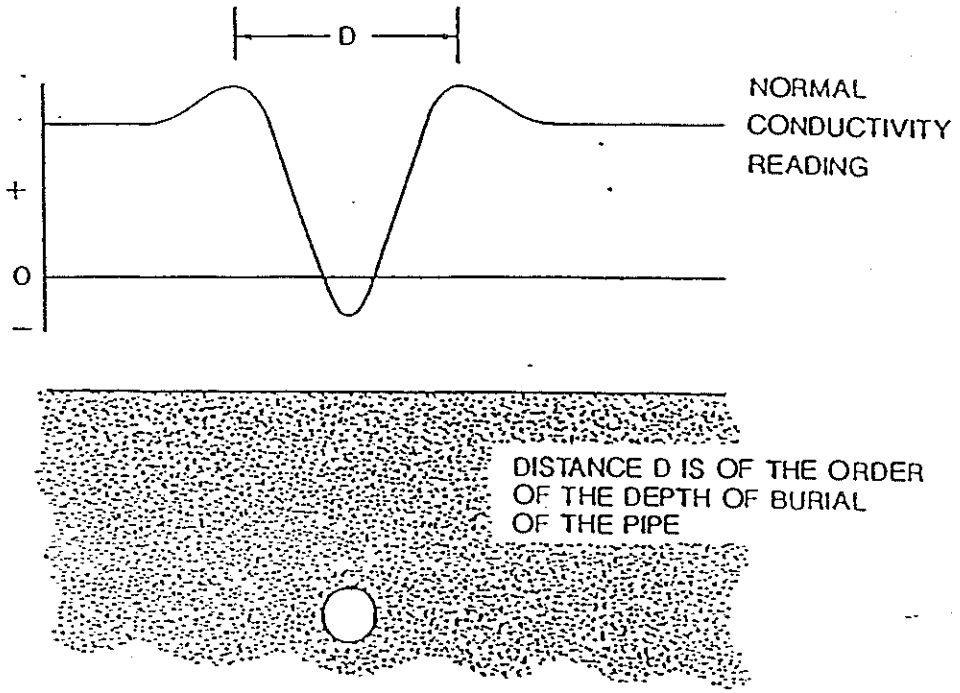





CONDUCTIVITY CONTOUR MAP

Figure 5






Source: Geonics Limited, Operating Manual for
EM31 Terrain Conductivity Meter, 1979

TYPICAL EM RESPONSE OVER A METAL PIPE

## 5.3 Equipment

The two-coil EM instrument and the VLF (very low frequency) instrument are basically the two different types of electromagnetic surveying instruments in use; each is capable of sensing to different depths. There are several models and manufacturers of this equipment.

The two-coil system consists of a transmitter coil and a receiver coil. Refer to Figure 1 which illustrates the basics of a two-coil electromagnetic induction apparatus. The transmitter coil induces an electromagnetic field of known strength and the receiver coil measures the resulting quadrature, or ratio of primary to secondary fields resulting from subsurface features. Each instrument is read directly in units of milliohms per meter (conductivity). EM readings represent the average bulk conductivity at a point halfway between the two coils.

The VLF instrument is a receiver which relies on specialized, very low frequency communication antennas for induction of an electromagnetic field. Surveying with the VLF or equivalent instrumentation is commonly referred to as VLF surveying.

The VLF Instrumentation is a small, lightweight hand-held instrument which can be operated by one person. Principal components of the instrument are a pair of mutually perpendicular coils and a receiving crystal with a frequency specific to a transmitting antenna. The two receiving coils are used to measure local characteristics of the primary induced field and any secondary fields emanating from bodies of variable conductivity. Typical sources of induced electromagnetic fields for VLF surveying are the very low frequency antennas used for submarine communications.

## 5.4 Data Acquisition

The advantage of the EM survey method is the speed and accuracy with which lateral changes of terrain conductivity can be measured. The EM conductivity data can be acquired using sounding and profiling techniques similar to those used in electrical resistivity. EM profiling is accomplished by traversing an area with a fixed coil spacing and orientation; EM sounding is accomplished by expanding the inter-coil spacings in a manner similar to that used by electrical resistivity soundings. Some commonly used EM equipment is limited in the number of available inter-coil spacings that can be used; however, there are other EM instruments available that can operate at many coil spacings and frequency ranges to provide numerous sounding data points necessary for accurate computer modeling and profiling.

The factors determining which instrument is used and what the grid spacing should be at particular sites are:

- Depth to target and size of target
- Accessibility of the site
- Effects of manmade structures and utilities, such as electric power lines
- Conductivity of the earth materials

EM induction instruments may have a depth of investigation of up to 200 feet depending upon coil spacing and orientations used (see Figure 3). The very low frequency VLF device has the greatest depth of investigation and is generally used to evaluate large geologic structures.

In conducting a VLF survey, VLF readings should be acquired with the instrument oriented perpendicular to a straight line from the site to the transmitter antennas. This orientation is necessary to ensure optimum data quality. All readings from a particular VLF station must be obtained with the instrument oriented in the same direction.

For an EM induction survey, a regular pattern of survey stations will provide coverage of the area in question. Typically, use of a grid spacing which is approximately equal to the size of the target sought by the survey, and a coil spacing with a maximum response for the depth of interest will produce satisfactory results. Specific needs for local detail, however, may require a refined coverage. The chosen spacing should always be site and target specific.

In conducting an EM survey, the field operator must avoid or note any potential sources of anomalous (noise) conductivity values such as power lines, buildings, fences, buried pipelines or any other large metal objects. Noise sources should be noted on the profiles or contour maps accounting for anomalies due to these known sources.

Important information that should be known for planning and before conducting an EM conductivity survey are: assumed hydrogeologic characteristics of the site, potential source locations and migration paths, characteristics of the hazardous substance of interest, and depths of interest. The level of detail necessary (size of object of interest and detail of resolution) determines the number of lines and station spacings of readings required.

EM data, if not recorded on a strip chart or digital recording instrument, should be recorded on standardized data sheets. At a minimum all data (strip chart, digital disks, or standard forms) should have the following information listed:

- Project/site location identification
- Company
- Date and time
- Operators name
- Instrument make, model
- Coil spacings and configuration
- Line and station numbers
- Instrument reading scales
- Weather conditions/temperature

## 5.5 Interpretation

### 5.5.1 Data Analysis

In general, electromagnetic survey data require relatively little processing before they can be interpreted. This is especially true for fixed coil spacing surveys because the data are recorded in units of conductivity; preliminary interpretations are made by comparison of conductivity values. A contour map can be prepared from the data and compared with results of other surveys. EM instruments also can be used for vertical soundings similar to resistivity sounding. Vertical sounding with EM equipment, however, has lower resolution than that performed with the resistivity technique. As a result, EM data are generally more useful for continuous profiling surveys.

VLF instruments do not read directly in units of conductivity. The in-phase measurement (the tilt of primary induced field) is read in terms of the tangent to the angle of tilt and is given as a percentage. Quadrature measurements, which are the ratios of voltage required to equalize the primary to secondary signal strengths, are also given as percentages. For field interpretation these two sets of data can be plotted in profile form, percentage versus distance. Greenhouse and Slaine (1983) describe a simple mathematical conversion so that VLF data can be presented in contour format and compared to other available data such as resistivity and magnetics. Digital data acquisition systems are now available that allow calculation of conductivity.

### 5.5.2 Presentation of Results

Results of an EM conductivity survey can be presented in profile and/or contour map form. The orientation of the traverses should be indicated on profiles in lines of coverage on contour maps. Locations of observed surface metal and other cultural features such as topography, buildings, fences, power lines etc. should be noted on both the profiles and the contour maps.

### 5.5.3 Interpretation

EM conductivity data can be analyzed qualitatively and quantitatively. Generally, profiling data are presented as a contour map or profiles. Profile lines should be stacked and aligned. A qualitative analysis of the contour map or aligned profiles usually can allow an interpreter to identify any conductivity trends that may be indicative of buried metal, groundwater flow and contaminant transport. A comparison of available geologic data, cultural ferrous metal and debris maps prepared during data acquisitions should be made to evaluate the causes of any conductivity trends observed.

Computer or chart comparisons of EM sounding data with available theoretical models can be made. This type of interpretation is similar to that used in electrical resistivity, but in EM sounding it is limited to relatively simple hydrogeologic conditions.

## 5.6 Advantages and Limitations

Advantages of the electromagnetic induction method include:

- No ground contact required
- Rapid data acquisition (faster than resistivity)
- Lightweight, one or two man operation
- Wide range of applications
- High lateral resolution
- Field interpretation possible

Limitations of the electromagnetic induction method include:

- Limited dynamic range 1 - 1,000 milliohm/meter
- Susceptible to effects of man-made structures, utilities, etc.
- Less vertical resolution than resistivity
- Limited penetration
- Does not distinguish even simple layering without more complex application and interpretation
- Setting and maintaining instrument at zero

## 6.0 QUALITY ASSURANCE RECORDS

Field data will be recorded in log books and/or data recording sheets accompanying the monitoring equipment. Data recorded in a Field Logbook will be entered with the following data: date, site location, personnel conducting the investigation, time (military time), start time and end time, weather.

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# F703
# *GROUND PENETRATING RADAR*

# GEOPHYSICS: GROUND PENETRATING RADAR (GPR)

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information and technical guidance on the methods and techniques of ground penetrating radar (GPR).

## 2.0 SCOPE

These procedures provide overall technical guidance.

## 3.0 DEFINITIONS

Bistatic Antenna – An antenna system in which transmitting and receiving coils are housed in separate antenna units.

Deconvolution – A computer processing method. The process of undoing the effect of another filter (in this instance the "earth"). A process that removes ringing, multiples, ghosts, and some background noise (Sheriff, 1973).

Dielectric Permittivity – (Also known as the relative dielectric permittivity): 1. A complex number consisting of a real and imaginary part, which uniquely describes the propagation and attenuation of electromagnetic energy in all materials. The real dielectric permittivity (dielectric constant) characterizes the propagation and reflection of electromagnetic (EM) waves, while the imaginary part (dielectric loss) characterizes the attenuation of EM signal (Kutrubes and Olhoeft, 1987). 2. A measure of the capacity of a material to store charge when an electric field is applied (Sheriff, 1973).

Electromagnetic Waves – One of the waves propagated by simultaneous periodic variations of electric and magnetic field intensity including radio waves, infrared, visible light, ultraviolet, X-rays and gamma rays (Webster's New Collegiate Dictionary. 1979).

Migration – Where velocity varies laterally, data will migrate (relative to the time versus antenna distance plot), and ray tracing is used to determine migrated positions (Sheriff, 1973).

## 4.0 RESPONSIBILITIES

Project Manager – The Project Manager is responsible for ensuring that the project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for ensuring that the personnel operating and interpreting the geophysical data are trained, skilled in that endeavor, so far as to receiving documentation on the training and experience of the operating personnel.

Field Team Leader – The Field Team Leader is responsible for selecting and detailing the geophysical technique and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the activities have been briefed and trained to execute these procedures.

## 5.0 PROCEDURES

### 5.1 Overview

Ground penetrating radar (GPR) is an active geophysical system which transmits high frequency electromagnetic (EM) waves into the ground and detects the energy reflected back to the surface. GPR operates on a similar principle as seismic reflection, except, instead of acoustic waves, electromagnetic waves of radio and microwave frequencies (80 MHz to 1,000 MHz) are utilized. Electromagnetic signals are reflected back to the surface from interfaces with differing electrical properties, such as dielectric permittivity and conductivity. The greater the contrast in the real dielectric permittivity (dielectric constant) between two materials, the more energy is reflected to the surface. Reflections typically occur at interfaces between lithological units with different characteristics, subsurface discontinuities, and internal structure such as:

- Top of bedrock surfaces
- Soil and rock stratification
- Water table
- Seepage and leachate zones
- Buried metal objects such as drums and utilities
- Open or water filled voids
- Bedrock fractures
- Archaeological structures

The depth of penetration of GPR is site specific, being limited by the attenuation of the electromagnetic energy. Signal attenuation is controlled by four different mechanisms listed below, any or all of which may be present at a site.

- Scattering losses
- Conduction losses
- Water losses
- Clay losses

Energy losses due to scattering occur when signals are dispersed in random directions, away from the receiving antenna, by large irregularly shaped objects, such as boulders and tree stumps.

Signal attenuation due to conduction is a function of the conductivity of a material, which varies with mineral composition, the amount of water, and the total dissolved solids (salts and heavy metals) within the water. The greater the electrical conductivity values of materials at a site, the more signal attenuation (hence less penetration) there will be.

Energy losses attributed to water occur when water molecules polarize in the presence of the applied electromagnetic field. Electromagnetic energy is lost to the radar system when it is converted to kinetic and thermal energy as a result of the rotation of water molecules.

Signal attenuation due to clay losses occurs when electrochemically charged ions polarize along clay surfaces in the presence of the electromagnetic field induced by the radar system. The migration and subsequent collision of these charged particles causes electromagnetic energy to be converted to kinetic and thermal energy, which is lost to the radar system.

Signal penetration is also dependent on the frequency of the transmitting antenna used in the radar system. Higher frequency antennas produce waves with shorter wave lengths, which are attenuated more rapidly with depth, but give better resolution. Specially designed 2 MHz antennas have been used to detect the ice-rock boundary of a 2 km thick glacier. Penetration of up to 75 feet has been reported for water saturated, clean sands in a Massachusetts glacial delta using a commercial antenna. Signal penetration in saturated clays, on the other hand, is on the order of magnitude of a few inches. Olhoeft (1986a) determined that even 5% clay added to a clean sand and gravel will cause a decrease in penetration by a factor of 20. Salt water is also a high loss substance, as signal penetration in sea water is less than a foot. It is important to note that in a layered medium a single, highly reflective layer alone can limit signal penetration by preventing the propagation of energy through it. In this instance the apparent loss of energy is caused by reflection rather than by signal attenuation.

## 5.2 Applications and Uses

Ground penetrating radar (GPR) is a shallow penetrating geophysical profiling system used for rapid and accurate surveys. GPR can be used for both area and source detection studies. GPR has been used to locate underground pipes, buried drums, foundations, voids in rock and concrete, lithologic contacts, to determine stratigraphy, depth to water table and depth to bedrock, and to locate buried archaeological artifacts, excavations, filled pits and lagoons, and numerous other site specific applications. GPR has been used successfully to delineate the lateral extent of plumes. Haeni et al. (1985) used GPR to investigate the thickness, type, and extent of sediments beneath a frozen lake with a 80 MHz antenna. The information acquired with GPR was used to help map the lateral extent of an aquiclude, and better estimate inputs to the mass balance equation for water budget calculations.

A GPR system can be used to determine depths to reflecting discontinuities by conducting a depth calibration. Typically, calibration is performed by moving the radar antenna over a metal target of known depth, such as a buried metal plate. Also, if transmitting and receiving antennas are housed in different units, designated as a bistatic antenna system, a common depth point (CDP) survey, similar to surveys conducted with seismic reflection, can be used to calculate the velocity of the medium, and hence depth to the reflector. Sakayama and others (1988) describe another method to calculate velocity from bistatic antennas where the receiving antenna is continually moved away from the stationary transmitting antenna. The velocities of the direct arrival and the first strong reflector are recalculated from the inverse slope of the time-distance display (antenna separation) on the GPR record in a similar manner as seismic refraction.

To verify GPR results, other geophysical or ground truth methods can be utilized. Haeni et al. (1985) utilized seismic refraction to correlate calculated depths of stratigraphic horizons and water tables with radar reflections. Magnetometry and electromagnetic induction methods have been utilized to verify the presence of buried drums and fuel tanks. Electromagnetic induction and electrical resistivity have been utilized to verify the lateral extent of conductive plumes. The depth to a particular reflector or target can also be verified by boreholes and/or test pit excavation.

## 5.3 Equipment

A typical ground penetrating radar system, shown on Figure 1, consists of:

- AC/DC power supply
- Control unit (pulse transmitter)
- Antenna(s)

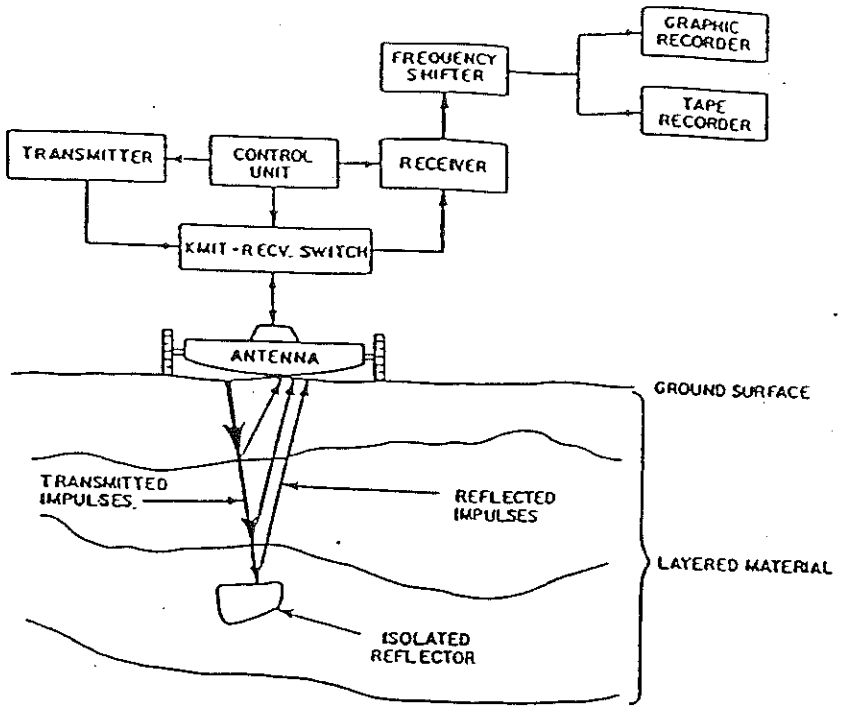
- Graphic recorder
- Digital recorder (optional)
- Magnetic tape recorder (optional)
- Coaxial cable which connects the control unit to the antenna

Typically, radar antennas contain both the transmitter and receiver within one fiberglass unit. Once a radar impulse is transmitted, the antenna switches to the receiver mode and records reflected radar impulses. The pulse receiver contains an amplifier which increase the amplitude of reflected signals. Bistatic antennas (transmitter-receiver are separate) allow the coverage of larger areas with one pass, and multi-receiver combinations allow the "stacking" of radar data which increases the signal to noise ratio.

Field data are generally printed by a graphic recorder and simultaneously can be stored on magnetic tape or diskette. The graphic recorder produces a continuous time (vertical) versus distance (horizontal) profile of the subsurface for field quality control and qualitative interpretations. Radar impulses are synchronized with the swept-stylus type graphic recorder, producing a dark band proportional to the amplitude of reflected radar signal. Because the antenna is moving, each pass of the stylus represents a slightly different antenna position. Gradually, as the recorder paper advances under the moving stylus, a pattern of reflective interfaces emerges. A typical radar record is shown on Figure 1.

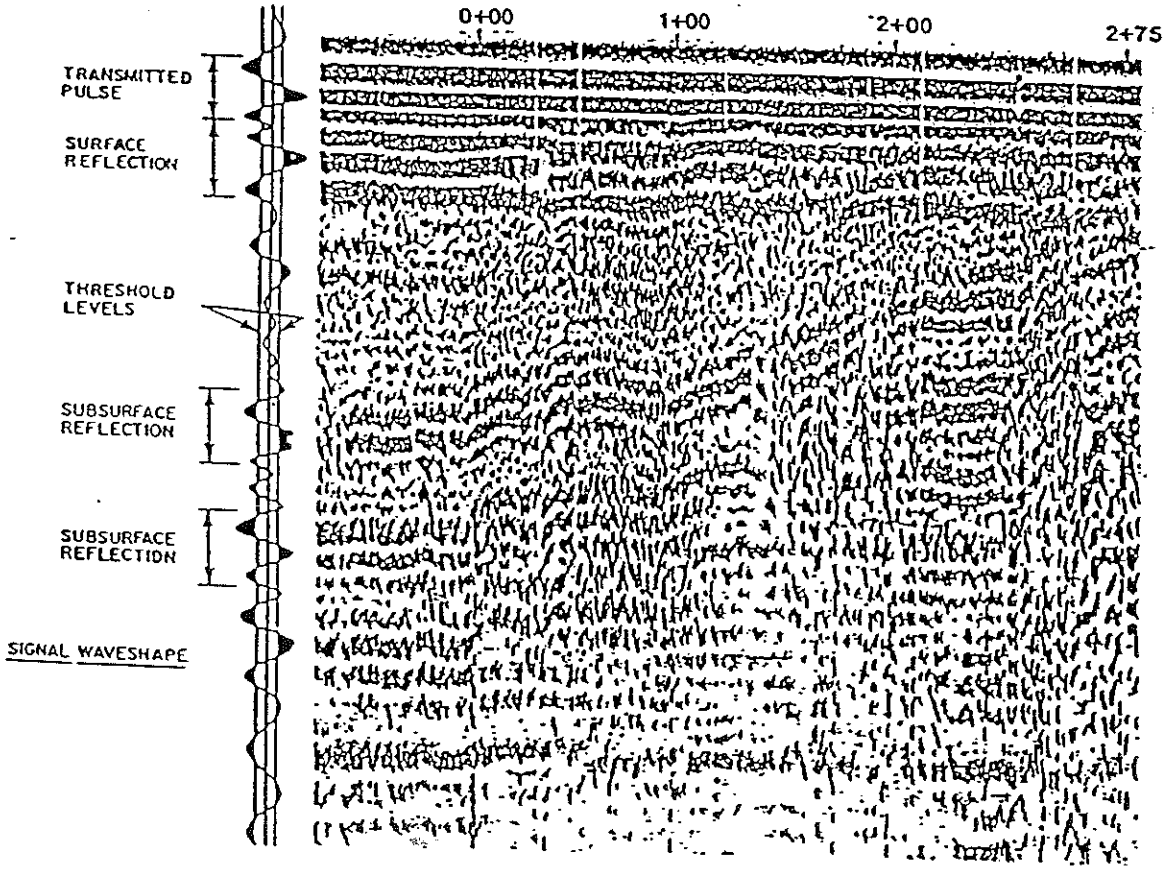
Storage of data on diskette or magnetic tape allows additional printing and/or computer processing for the refinement of data. Deconvolution of stored data enhances stratigraphic reflections from the water table and soil structures (Olhoeft, 1988). Migration of data allows easier resolution of metallic targets, such as buried drums, and delineation of excavations and sinkholes (Hogan, 1988).

Radar systems are designed to use antennas of various electrical characteristics. Selection of the antenna is dictated by the requirements of the survey. If high resolution, near-surface data are desired, a small, high frequency antenna is used; if the survey requires deeper probing, a larger, lower frequency antenna is used (80, 120, 250, 300, 400, 500, 900, and 1,000 MHz antennas are commercially available). The drawback of using the lower frequency antennas is that resolution of data is sacrificed for penetration. Also, the low frequency antennas (less than 250 MHz) are generally not shielded, making them susceptible to overhead power line noise and spurious reflections from passing cars. The 900 and 1,000 MHz antennas are used almost exclusively for shallow penetration projects such as the detection of rebar in concrete, as their penetration is generally limited to 2 to 3 feet.




RADAR SYSTEM BLOCK DIAGRAM

FIGURE 1




TYPICAL RADAR RECORD

GROUND PENETRATING RADAR SET-UP

## 5.4 Field Procedures

The majority of time involved with any GPR survey is spent establishing survey lines in the area of investigation so that detected anomalies can be easily located. Survey lines should be set to maximize coverage, while maintaining a grid spacing proportional to the presumed target dimensions. A minimum survey line spacing of 10 feet is desired when looking for a 1,000 gallon fuel tank, while a larger spacing of 50 feet or more may be used to define the lateral extent of a conductive plume.

At the onset of any GPR survey the radar control unit should be adjusted for the anticipated depth of penetration. Adjustments of the time window of exploration should be made by estimating the velocity of the medium and desired depth of penetration. Most surveys can be effectively conducted with a minimum time window of 50 nanoseconds.

Accurate determination of the depth to any layer requires calibration of the radar system. The simplest way of calibrating the GPR system to specific settings is by burying a plate at a measured depth, and moving the antenna slowly along the survey line. The plate will produce on the GPR record a thick, dark band, parabolic or flat in shape, with many multiple reflections beneath it. Once a certain confidence level is attained from depth calibration, the survey is conducted by slowly pulling the antenna along survey lines. A slow walking pace increases the horizontal resolution as radar signals are propagated in a 15 to 45 degree cone from the bottom of the antenna. A slow walking pace is recommended for identifying buried objects or plumes as targets are better defined and easier to resolve. On the other hand, the radar antenna can be towed from the back of a car or truck at speeds up to 10 miles an hour if the "target" is a continuous reflector, such as the water table.

## 5.5 Interpretation

A typical GPR record is shown on Figure 1. A representation of a single GPR signal pulse is shown along the side of the record. The horizontal scale of the record is maintained by marking on the record the locations of survey stations as they are reached by the antenna. Accurate determination of the vertical scale (i.e., conversion of a time into a depth) requires calibration of the radar system. If the depth to a known reflector can not be determined through calibration or verification using boreholes and test pits, the velocity of the medium can be approximated from relationships involving the velocity of the medium, and the dielectric constant (real dielectric permittivity) of the medium. Values of the dielectric constant can be found in GSSI (1974), and Kutrubes (1986). It is important to note that the relationship is not valid when signal losses are great. The estimated depth to the reflector can be calculated from time and velocity values.

Interpretation of GPR data is subjective, even among experienced interpreters. The strength of a reflected signal and/or the continuity of the reflector across the record may be indicative of a stratigraphic contact. The water table in an unconfined sand and gravel aquifer may also produce a similar signature on the GPR record. The strong continuous reflector shown in Figure 2 delineates the contact between bedrock and unconsolidated materials. Point targets, such as buried drums, pipes, boulders, tree stumps, etc., create a distinctive parabolic feature on GPR records. Positive identification of point targets is subjective, as the GPR signature of a pipe is similar to that of a large boulder.

Figure 3 shows the characteristic parabolic signal created by underground storage tanks situated in a clean sand and gravel deposit. Metallic objects, such as buried drums and pipes, also produce a characteristic parabolic signal on the record, and sometimes produces a "ringing noise," denoted by the heavy, dark banding, as shown in Figure 3.

Recorded data from diskettes or magnetic tapes can be enhanced using numerous computer processing methods to remove constant noise problems, such as ringing, or to sharpen up geologic contact features and point target boundaries (such as underground storage tanks). Such methods are described in detail by Hogan (1988) and Olhoeft (1988). Computer processing is costly and generally not necessary in most instances.

## 5.6 Advantages/Disadvantages

Advantages of ground penetrating radar systems include:

- Rapid area coverage
- Not destructive
- Portable equipment
- High vertical resolution profiles in the field for immediate interpretation

Ground penetrating radar provides a cost effective way of evaluating a large site in a short amount of time. One day of work coverage completed by GPR may be equivalent (with respect to data generated) to 4 or 5 days of work with seismic refraction or electrical resistivity. The GPR method is "non-destructive" in that it does not necessarily require excavation or probing of the overburden materials, although verification of anomalies should be conducted.

Equipment can be easily loaded in the back of a truck as most pieces comprising the radar system weigh under 40 pounds. The 80 MHz antenna weighs about 100 pounds and is less portable as it is approximately 4 feet wide.

Limitations of radar systems include:

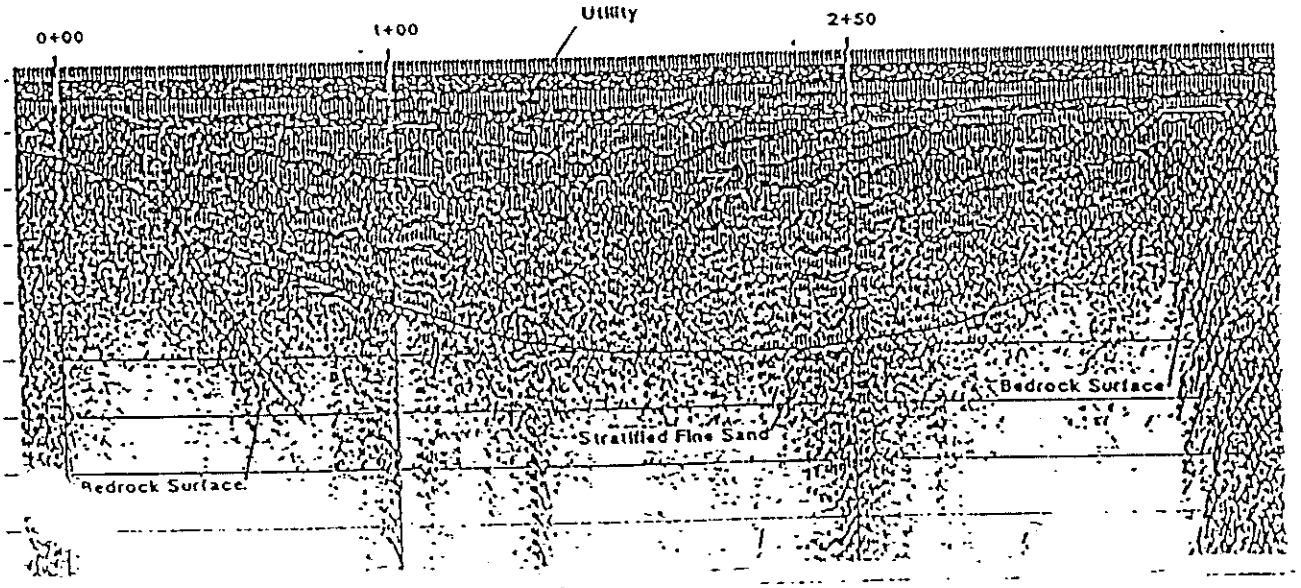
- Survey lines must be cleared to ground level
- Multiple receiver antennas are generally required to stack and process radar data
- Penetration is site specific, requiring data corroboration using alternative geophysical methods and/or verification
- Interpretations are subjective

To maximize resolution and minimize scattering losses, survey lines must be as level as possible to prevent the bouncing and jarring of the radar antenna. Survey lines cleared of debris also allow the antenna to be pulled at an even, continuous pace, permitting more accurate determination of horizontal scale.

Application of GPR is limited by soil type and presence of high loss materials. Delineation of buried objects beneath conductive plumes also may not be possible. The unpredictability of radar effectiveness requires that site investigations be conducted with alternative geophysical methods, such as electromagnetic induction, seismic refraction, magnetometry, and/or electrical resistivity.

FIGURE 2

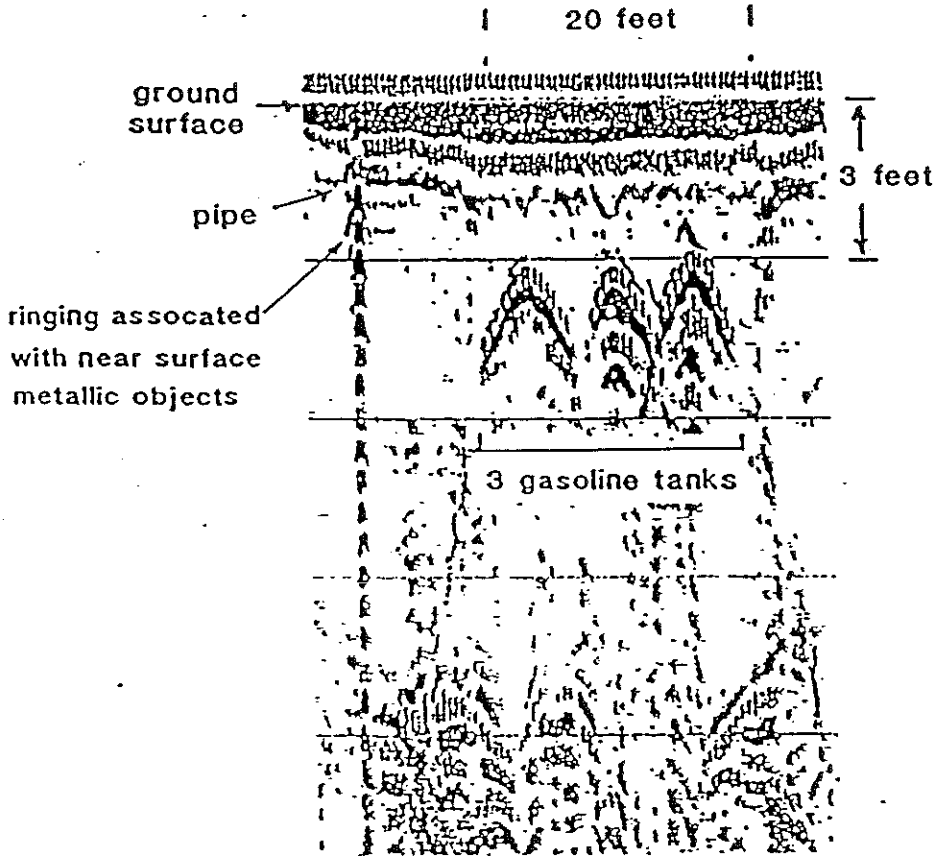





GROUND PENETRATING RADAR RECORD
OF A BURIED RIVER CHANNEL

FIGURE 3






GROUND PENETRATING RADAR RECORD
OF BURIED FUEL TANKS

## 6.0 QUALITY ASSURANCE RECORDS

All data will be recorded in Field Logbooks with the following information: date, location, personnel on site, start and finish times, equipment, instrument gain and time settings, project number, site conditions, and weather.

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# F704
# *MAGNETOMETRY*

# GEOPHYSICS: MAGNETOMETRY

## 1.0 PURPOSE

This SOP provides general reference information and standard techniques for using magnetometry.

## 2.0 SCOPE

This SOP provides a description of the field procedures, equipment, and interpretation methods necessary to fully utilize this procedure.

## 3.0 DEFINITIONS

Diurnal variations - daily changes in the total magnetic field strength due to solar activity which may exceed 100 gammas.

Gradient - change in magnetic field strength over a given vertical or horizontal distance.

Magnetic storm - sudden and simultaneous variations of up to several hundred gammas throughout the world. Magnetic storms may occur several times a month and last several days.

Total magnetic field intensity - a scaler measurement (independent of direction) of the magnitude of the earth's magnetic field.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that the project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for ensuring that the personnel operating and interpreting the geophysical data are trained, skilled in that endeavor, so far as to receiving documentation on the training and experience of the operating personnel.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the geophysical technique and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the activities have been briefed and trained to execute these procedures.

## 5.0 PROCEDURES

### 5.1 Overview

Magnetic surveying is a passive geophysical technique which measures the strength of the earth's magnetic field. The earth's field is a vector quantity having a unique magnitude and direction at every point on the earth's surface. A magnetometer is the instrument which measures the magnetic field strength in units of gammas or nanoteslas. In order to recognize a magnetic anomaly, it must be several times larger than the background noise level along that profile. Buried ferrous metal objects such as steel drums or tanks cause

local variations or anomalies in the earth's magnetic field that can be detected by a magnetometer. Geologic features such as igneous intrusion or iron rich sands can also be mapped using magnetic surveying.

The earth's magnetic field is not completely stable. It undergoes long-term (secular) variations over centuries; small, daily (diurnal) variations (less than 1% of the total field magnitude); and transient fluctuations (magnetic storms) resulting from solar flare phenomena. Both naturally occurring and manmade magnetic materials can modify the earth's magnetic field locally.

Analysis of magnetic data by an experienced geophysicist can provide an estimate of the areal extent and quantity of buried ferrous objects. Depth of burial approximations can be made using graphical methods of interpretation such as slope techniques and half-width rules as described in Nettleton (1976).

## 5.2 Application and Uses

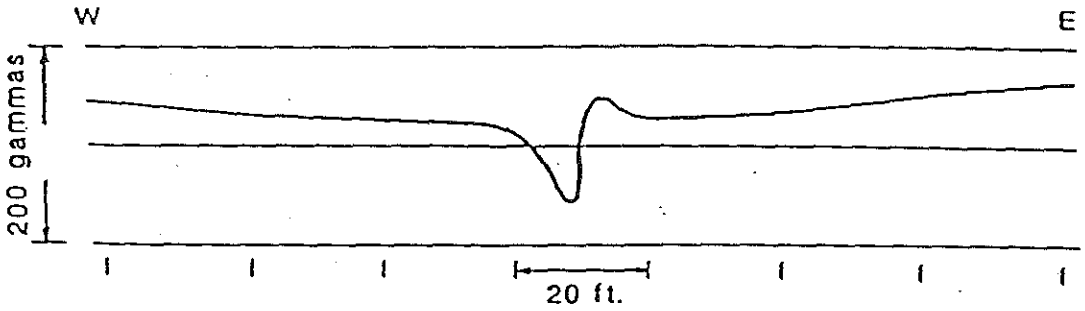
Buried ferrous metal objects such as pipelines, barrels, tanks, etc., generally produce a perturbation in the earth's naturally occurring magnetic field. The size (amplitude) of this perturbation is related to the size of, distance to, susceptibility and remanent magnetization of the buried object. The magnetic survey method, therefore, is a useful tool for site studies to locate and identify buried ferrous metal. Figures 1 and 2 show magnetic profiles and contour maps of areas containing buried metal objects. Nonanomalous magnetic data acquired where EM conductivity anomalies exist indicate the existence buried of conductive, nonferrous metal (copper, aluminum, brass) objects.

Magnetic data can be helpful in determining the size and geometry of geologic features such as fault zones, mineralized zones, and bedrock valleys and depressions. These features generally are characterized by longer wavelength anomalies and are readily distinguishable from anomalies associated with buried metal. In many areas, such geologic features may control or influence the direction and velocity of groundwater flow.

## 5.3 Equipment

Magnetometers commonly used in hazardous waste site investigations include the total field proton precession magnetometer, the flux gate magnetometer, and the magnetic gradiometer. Text books such as Telford (1976) and Nettleton (1976) discuss in detail the operation and construction of these and other magnetometers.

FIGURE 1

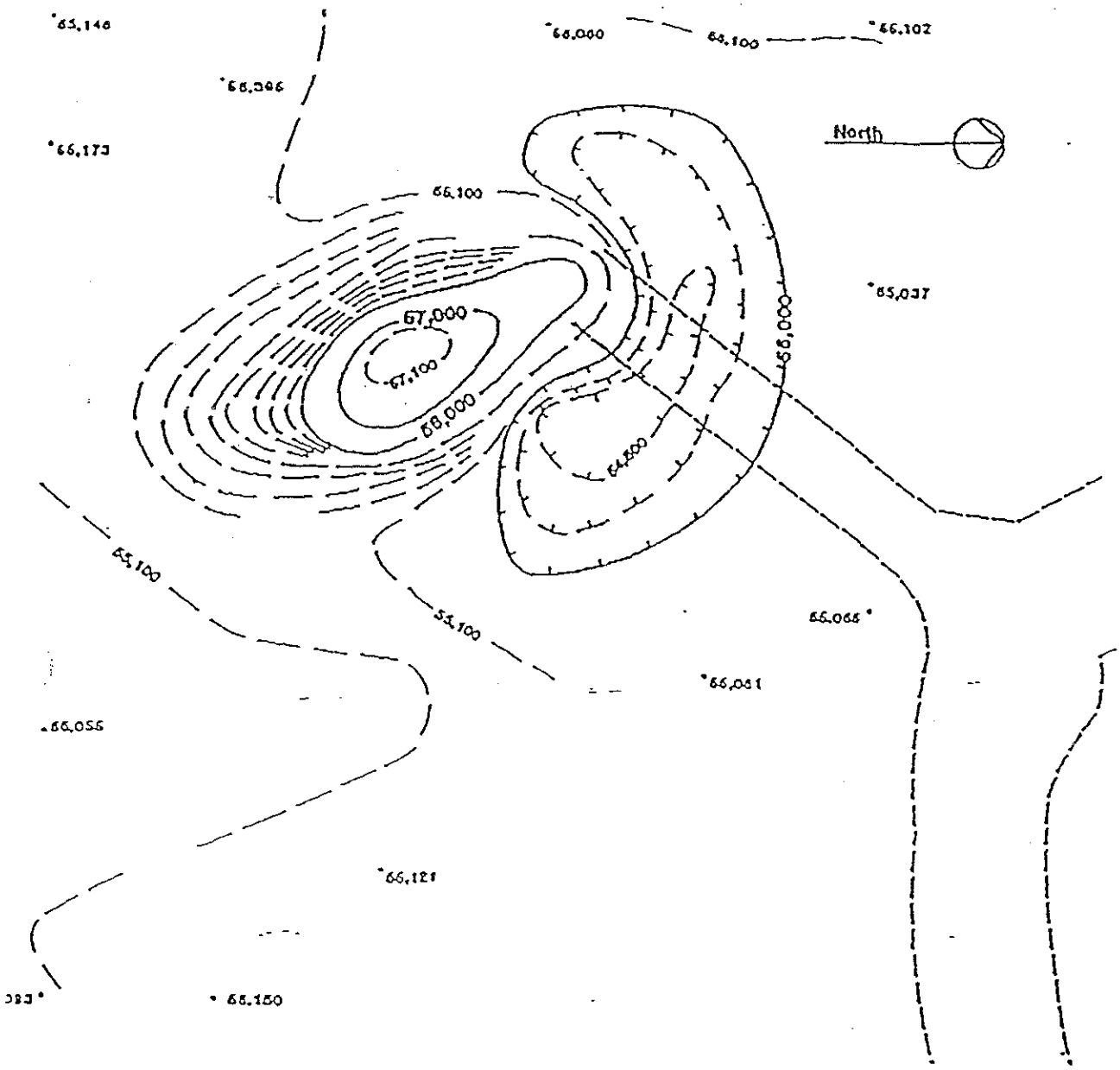



MAGNETIC ANOMALY FROM A SINGLE BARREL
BURIED AT A DEPTH OF 6-7 FT.

MAGNETIC ANOMALY FROM A BURIED BARREL

FIGURE 2






55,000 Magnetic contours (gammas)

66,103 Spot magnetic value (gammas)

0 20 40

Scale in Feet

MAGNETIC ANOMALY CAUSED BY 75 BARRELS BURIED 4 TO 10 FEET DEEP

The total field proton precession magnetometer is the most commonly used magnetometer in hazardous waste investigations. This instrument utilizes the precession of spinning protons of hydrogen atoms in a sample fluid (kerosene, alcohol, or water) to measure the total magnetic field intensity. Total field proton precession magnetometers are portable and do not require precise orientation and leveling; the sensor must be oriented with one side facing approximately north and the sensor held stationary during the cycling period. Proton precession magnetometers have no instrument drift, do not require calibrations, are easy to operate, and have an accuracy of 0.1 gamma. Most modern proton precession magnetometers have digital readouts and electronic storage of data.

Vertical magnetic gradiometers are magnetometers that measure vertical differences of the earth's total magnetic field. Gradient measurements enhance magnetic anomalies resulting from near surface magnetic source and discrimination between neighboring magnetic anomalies is also enhanced. These measurements are generally made using an instrument similar to a total field magnetometer that has two or more sensors mounted on a staff. The sensors are vertically separated by a constant distance, usually one to three feet. Gradient readings are adversely affected by ferrous metal surface debris since signals from this surface debris are also amplified. Consequently, removal of surface metal should be considered before conducting a gradiometer survey.

The flux gate magnetometer was developed during World War II as a submarine detector. Text books such as Telford (1976), RAO and Murthy (1978) explain in detail the principals of operation of the flux gate magnetometer. A fluxgate magnetometer can define the boundaries of regions of buried ferrous metal objects more precisely than the proton precession magnetometer. There are several sources of errors in flux gate magnetometers including unbalance in the two coils, thermal and shock noise, circuit drift and temperature sensitivity. The advantages are direct readout, no azimuth orientation, only coarse leveling required, light weight and portability (Telford, 1976).

## 5.4 Field Procedures

Magnetic data are generally acquired at relatively close station spacings (5 to 50 foot intervals) along closely spaced (10 to 50 feet) parallel survey lines.

Magnetic data can be acquired in a rectangular grid pattern or along traverses. Grid data are readings acquired at the nodes of a rectangular grid; traverse data is acquired at fixed intervals along a line. Traverse data is often preferable to grid data because it generally is less expensive to acquire (heavily vegetated sites require time-consuming brush cutting to establish a complete grid) and more useful for interpretation than an equal number of grid readings. Traverse lines generally should be oriented in a north-south direction so that the maximum amplitude of an anomaly can be detected. However, line orientations are often dependent on site obstructions and sources of magnetic noise.

Station and line spacing intervals are determined on the basis of the desired resolution of the survey. If individual drums or clusters of deeply (greater than 25 feet) buried drums are the objective of the survey, then a detailed magnetic survey with relatively close station spacings (approximately 5 to 10 feet) and line spacings (approximately 10 to 25 feet) should be used. If large metal objects such as 10,000 gallon tanks or trenches filled with barrels are the objective of the magnetic survey, then a reconnaissance or screening survey with larger station spacings (25, 50, or 100 feet) and line spacings of (25, 50, or 100 feet) may be appropriate.

In conducting a survey, the field operator must avoid or note any apparent sources of high magnetic gradients and alternating currents, such as power lines, buildings, and any large iron or steel objects. It is also important that the operator be relatively free of magnetic materials on his/her person and the magnetometer sensor be kept clean to avoid possible magnetic particles. Periodically during a survey, and particularly when an anomaly is detected, it is important to establish that the magnetometer is providing valid readings and not random, meaningless instrument noise. The simplest means of verifying magnetometer field readings is to take several successive readings at one location. These readings should repeat to within ±1 gamma. Readings are taken at predetermined intervals which depend on the nature of the survey and which may have to be modified depending on the gradients encountered. For detailed surveys, a base station or a set of stations occupied several times per day, or a continuous monitoring station (within 100 miles) is established to check for diurnal variations and magnetic storms. At the height of a magnetic storm, magnetic surveying may be impractical due to the large instantaneous changes in the total magnetic field.

## 5.5 Interpretation

### 5.5.1 Data Analysis

Magnetic data can be corrected for diurnal variations; however, diurnal changes are generally very gradual and linear and should not have the extreme fluctuations associated with buried ferrous metal objects. Magnetic data can be plotted in profile form or contoured depending upon the survey coverage. Noise sources (surface ferrous metal objects, fences, power lines, etc.) should be noted on the profiles or contour map so that anomalies due to these known sources can be accounted for. The amplitudes of similar sized surface metal objects should be compared. If similar sized ferrous metal surface objects have extremely different anomaly amplitudes, it may be an indication that buried ferrous metal objects exist in the vicinity of the higher amplitude anomalies.

### 5.5.2 Presentation of Results

The results of a magnetic survey should be presented in profile and/or contour map form. The orientation of the traverses should be indicated on profiles and lines of coverage on contour maps. Locations of observed ferrous metal and other cultural features (hills, valleys, streams, etc.) should be noted on both the profile and the contour maps.

### 5.5.3 Interpretation

Magnetic anomalies can be analyzed both qualitatively and quantitatively. The shape and gradient of an anomaly (slope, wave-length, amplitude, etc.) contains enough information to draw qualitative conclusions regarding the location and depth of the causative source.

Quantitative computer modeling interpretations of magnetic data are complicated both by the inherent complexity of dipole magnetic behavior and by the fact that a number of different types and configurations of sources can cause the same anomaly. Where the properties of the earth's field and the local geologic materials (inclination, declination, susceptibility, and remanent magnetization) are well known, reasonable assumptions regarding the nature of the source can be made, and a fairly accurate model of the source generally can be derived.

## 5.6 Advantages and Limitations

Advantages of the magnetic survey method include:

- Rapid operation
- Low expense
- Identification of buried metal (ferrous)
- Sensitivity to small ferrous objects

Limitations of the magnetic survey method include:

- Susceptible to effects of manmade structures, utilities, buildings, fences, etc.
- Detection is limited to the distance to and quantity of ferrous metal present

## 6.0 QUALITY ASSURANCE RECORDS

All data will be recorded in Field Logbooks and/or data logging sheets designed for this procedure. All data will be entered with the following basic information: date, start and end times, location, personnel on site, equipment, project number, site conditions, and weather.

## 7.0 REFERENCES

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# F705
# *SEISMIC METHODS*

# GEOPHYSICS: SEISMIC METHODS

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information for using seismic methods.

## 2.0 SCOPE

This SOP indicates the normal methods and techniques that may be used and the manner for interpretation of seismic data.

## 3.0 DEFINITIONS

Geophone - Vibration sensitive detectors.

Hydrophones - Pressure sensitive detectors.

Reflection - The returned energy from a shot or other seismic source which has been reflected from an acoustic-impedance contrast.

Refraction - The deflection of the direction of a wave propagation when waves pass obliquely from one velocity material to another.

Shot points - Origin of shock waves.

Snell's Law - Law of refraction.

Zero time - Exact moment of shock wave origin.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that the project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for ensuring that the personnel operating and interpreting the geophysical data are trained, skilled in that endeavor, so far as to receiving documentation on the training and experience of the operating personnel.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the geophysical technique and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the activities have been briefed and trained to execute these procedures.

## 5.0 PROCEDURES

### 5.1 Overview

Seismic exploration methods utilize the natural energy transmitting properties of soils and rocks and are based on the principle that the velocity at which seismic waves travel through the earth is a function of the physical properties (elastic moduli and Poisson's ratio) of the materials. Interpretations are based on the time required for a seismic wave to travel from a source to a series of vibration sensitive detectors (geophones) located at specific intervals along the ground surface. The resultant seismic velocities are used for:

- Material identification.
- Stratigraphic correlation.
- Depth determinations.
- Calculation of elastic moduli values and Poisson's ratio

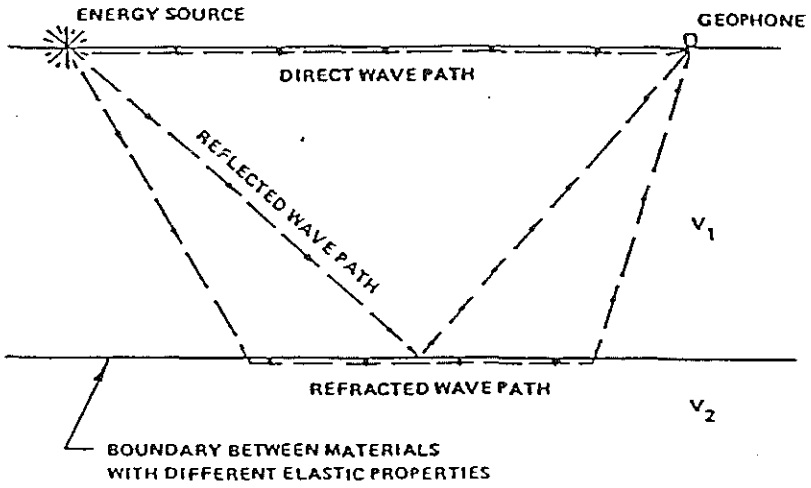
A variety of seismic wave types, differing in resultant particle motion, are generated by a near surface seismic energy source. The two types of seismic waves for seismic exploration are the compressional (P) wave and the shear (S) wave. Particle motion resulting from a P-wave is an oscillation, consisting of alternating compression and dilatation, oriented parallel to the direction of propagation. An S-wave causes particle motion transverse to the direction of propagation. The P-wave travels with the higher velocity of the two waves and is of greater importance for seismic surveying. The following discussions are concerned principally with P-waves.

Possible seismic wave paths include a direct wave path, a reflected wave path or a refracted wave path. These wave paths are illustrated in Figure 1. The different paths result in different travel times, so that the recorded seismic wave form will theoretically show three distinct wave arrivals. As one would expect, the direct and refracted wave paths are important to seismic refraction exploration while the reflected wave path is important for seismic reflection studies.

Seismic waves incident on the interface between materials of different elastic properties at what is termed the critical angle are refracted and travel along the top of the lower layer. The critical angle is a function of the seismic velocities of the two materials. These same waves are then refracted back to the surface at the same angle. The recorded arrival times of these refracted waves, because they depend on the properties and geometry of the subsurface, can be analyzed to produce a vertical profile of the subsurface. Information such as the number, thickness and depths of stratigraphic layers, as well as clues to the composition of these units can be ascertained.

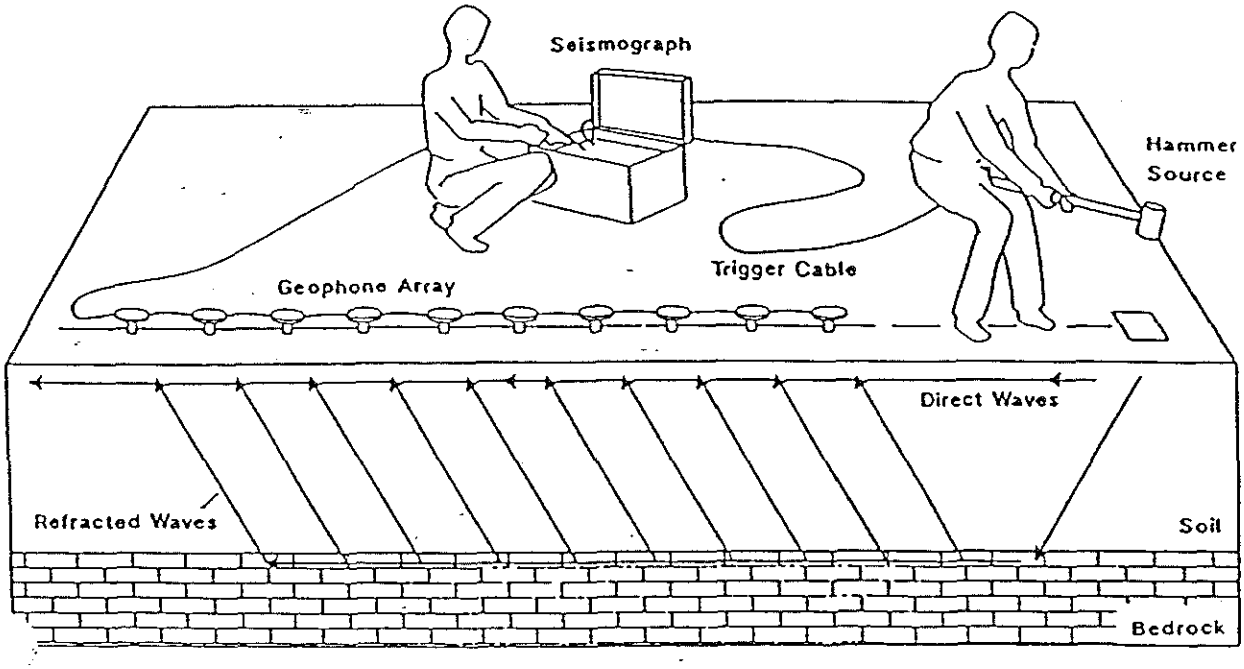
The first arrivals at the geophones located very near the energy source are direct waves that travel through the near surface. At greater distances, the first arrival is a refracted wave as illustrated in Figure 2. Lower layers typically are higher velocity materials, therefore the refracted wave will overtake both the direct wave and the reflected wave, because of the time gained traveling through the higher velocity material compensates for the longer wave path. Depth computations are based on the ratio of layer velocities and the distance from the energy source to the point where refracted wave arrivals overtake direct arrivals. Only reflected waves will be produced, when the angle of incidence is vertical or exceeds the critical angle.

Figure 1




SEISMIC WAVE PATHS FOR DIRECT WAVE, REFLECTED WAVE, AND REFRACTED WAVE ILLUSTRATING EFFECTS OF A BOUNDARY BETWEEN MATERIALS WITH DIFFERENT ELASTIC PROPERTIES

Figure 2




Source: Benson, Glaccum, and Noel (1982)

FIELD LAYOUT OF A 12-CHANNEL SEISMOGRAPH SHOWING THE PATH OF DIRECT AND REFRACTED SEISMIC WAVES IN A TWO-LAYER SOIL/ROCK SYSTEM

A constraint on refraction theory is that material velocities ideally should increase with depth. Although not the usual case, if a velocity inversion exists, i.e. where a low velocity layer is overlain by a higher velocity layer, depths and seismic velocities can be calculated but the uncertainty in calculations is increased unless borehole data are available.

The basis for seismic reflection surveying is the time required for a seismic wave to travel from the source, to a discrete reflector interface and for the reflected wave to return to the surface (two-way travel time). Both the energy of the reflected wave and the diagnostic wave form are a function of acoustic impedance contrast across the interface. Acoustic impedance characteristics of a material depend on seismic velocity and density.

More rigorous discussions of seismic wave theory as applied to seismic reflection and refraction can be found in Dobrin (1976), Telford and others (1976), Griffiths and King (1981), and Mooney (1977).

## 5.2 Applications

The seismic refraction technique is an accurate and effective method for determining the thickness of subsurface geologic layers. Applications for groundwater and hydrogeologic studies include:

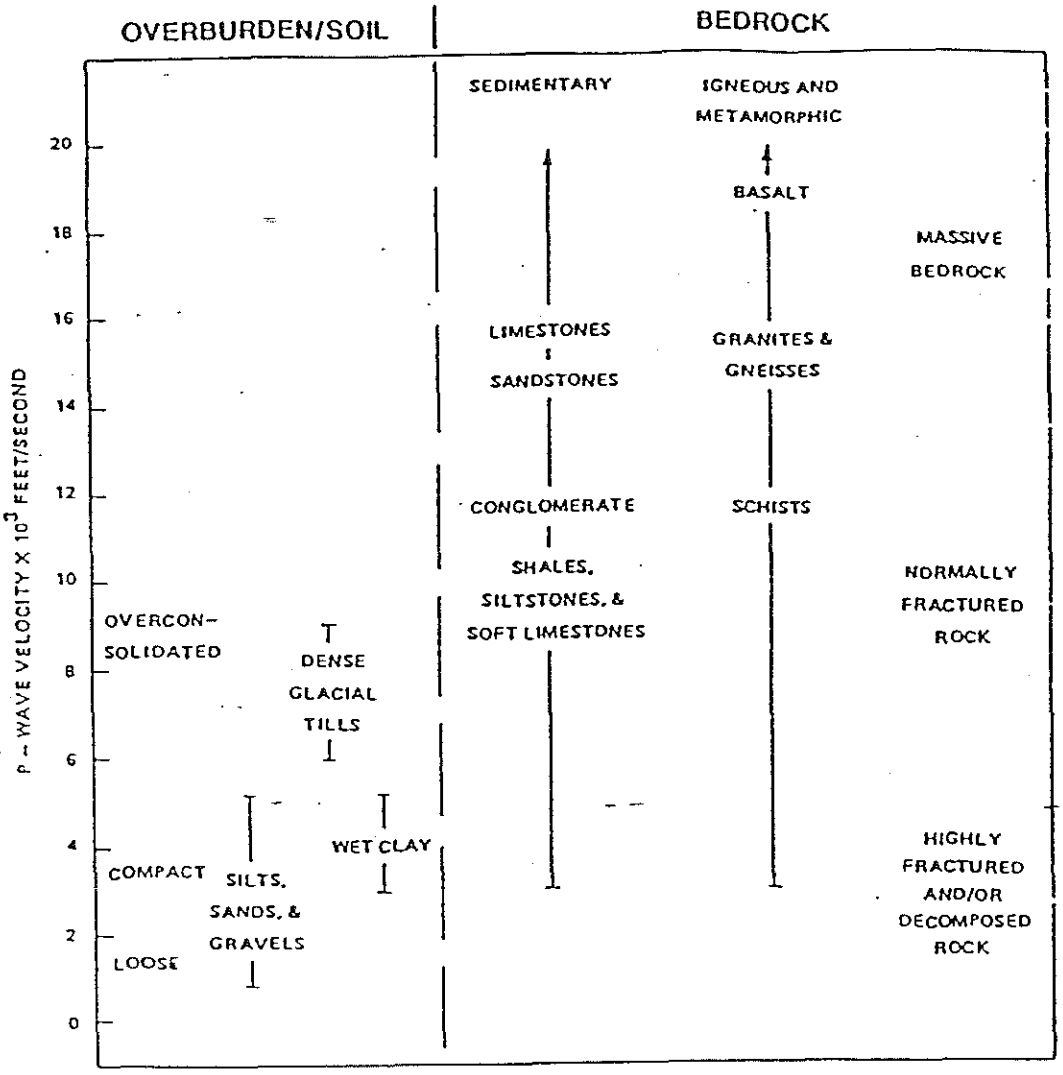
- Continuous profiling of subsurface layers including the bedrock surface;
- Water-table depth determinations;
- Mapping and general identification of significant stratigraphic layers;
- Detection of sinkholes and cavities;
- Detection of bedrock fracture zones; and,
- Detection of filled-in areas.

Seismic refraction investigations are particularly useful because seismic velocities can be used for material identification. Figure 3 presents a guide to material identification based on P-wave seismic velocities. In rocks and compacted overburden materials, the seismic waves travel from grain to grain so that the measured seismic velocity value is a direct function of the solid material. In porous or fractured rock and most overburden materials the seismic waves travel partly or wholly through the fluid between the grains.

Seismic compressional wave velocities in unconsolidated deposits are significantly affected by water saturation. The seismic velocity values of unsaturated overburden materials such as gravels, sands and silts generally fall in the range of 1,000 to 2,000 ft/sec. When these materials are water saturated, that is when the space between individual grains are 100% filled with water, the seismic velocities range from 4,800 to 5,100 ft/sec, equivalent to the compressional P-wave velocity of sound in water. This is because the seismic wave assumes the velocity of the faster medium, that of water. Even a small decrease in the saturation level will substantially lower the measured P-wave velocity of the material. Because of this velocity contrast between saturated and unsaturated materials, the water table acts as a strong refractor.

Figure 3






GUIDE TO MATERIAL IDENTIFICATION
BY P-WAVE SEIMIC VELOCITY

Seismic investigations over unconsolidated deposits are used to map stratigraphic discontinuities and to unravel the gross stratigraphy of the subsurface. These can be vertically as in the case of a dense till layer beneath a layer of saturated material or horizontally as in the case of the boundaries of a fill material. Often these boundaries represent significant hydrologic boundaries, such as those between aquifers and aquicludes.

A common use of seismic refraction is the determination of the thickness of a saturated layer in unconsolidated sediments and the depth to relatively impermeable bedrock or dense glacial till (see Figure 3). Continuous subsurface profiles and even contour maps on the top of a particular horizon or layer of interest can be developed from a suite of seismic refraction data.

Bedrock velocities vary over a broad range depending on variables which include:

- Rock type
- Density
- Degree of jointing/fracturing (and fracture saturation for compressional waves)
- Degree of weathering

Fracturing and weathering generally reduce seismic velocity values in bedrock. Low velocity zones in seismic data must be evaluated carefully to determine if they are due to overburden conditions or fractured/weathered or perhaps even faulted bedrock.

Seismic reflection surveys are generally used for greater depths of investigation (hundreds of feet). High resolution shallow reflection surveys have had some limited success in the upper few hundred feet but only under ideal conditions (flat surface topography and subsurface layering) and are not considered generally applicable to resolving the data objectives of shallow seismic investigations. In particular, the reflection technique does not directly measure seismic velocities, a necessary element to interpreting subsurface seismic data from a hydrogeologic viewpoint.

## 5.3 Equipment

The basic equipment necessary to conduct a seismic refraction investigation consists of:

- Energy source
- Seismometers (Geophones)
- Seismic cables
- Amplifier
- Recording unit

Geophones are sensitive vibration detectors which convert ground motion to an electric voltage for recording the seismic wave arrivals. Seismic cables, which link the geophones and amplifier, are generally fabricated with pre-measured locations for geophones and shot points definitions. The amplifier increases the voltage output of the geophones and is capable of selectively filtering the signal.

Recording of seismic data is conducted in either analog or digital formats with single or multichannel recording equipment. Multichannel data acquisition systems (12 or 24 channel) are much preferred and necessary for all but the simplest of very shallow surveys. In general, the greater the number of channels the more data is available for analysis which results in higher resolution of seismic velocities and depth determinations. Analog records are paper prints of the geophone response to seismic wave arrivals from

which the travel time between the shot and the first arrival signals for each geophone can be measured directly. Figure 4 is an example of an analog record showing one recorded trace for each geophone, vertical timing lines, zero-time break and the first arrival signals at each geophone. A magnetic media recorder is required for digital recording. Digital recording and subsequent computer processing enable more extensive and detailed interpretation of seismic data.

Energy sources used for seismic surveys are categorized as either non-explosive or explosive. The energy for a non-explosive seismic signal can be provided by one of the following:

- Airgun
- Seisguns
- Weight drop
- Sparker
- Sledgehammer (very shallow penetration)
- Vibrators (for reflection surveys)

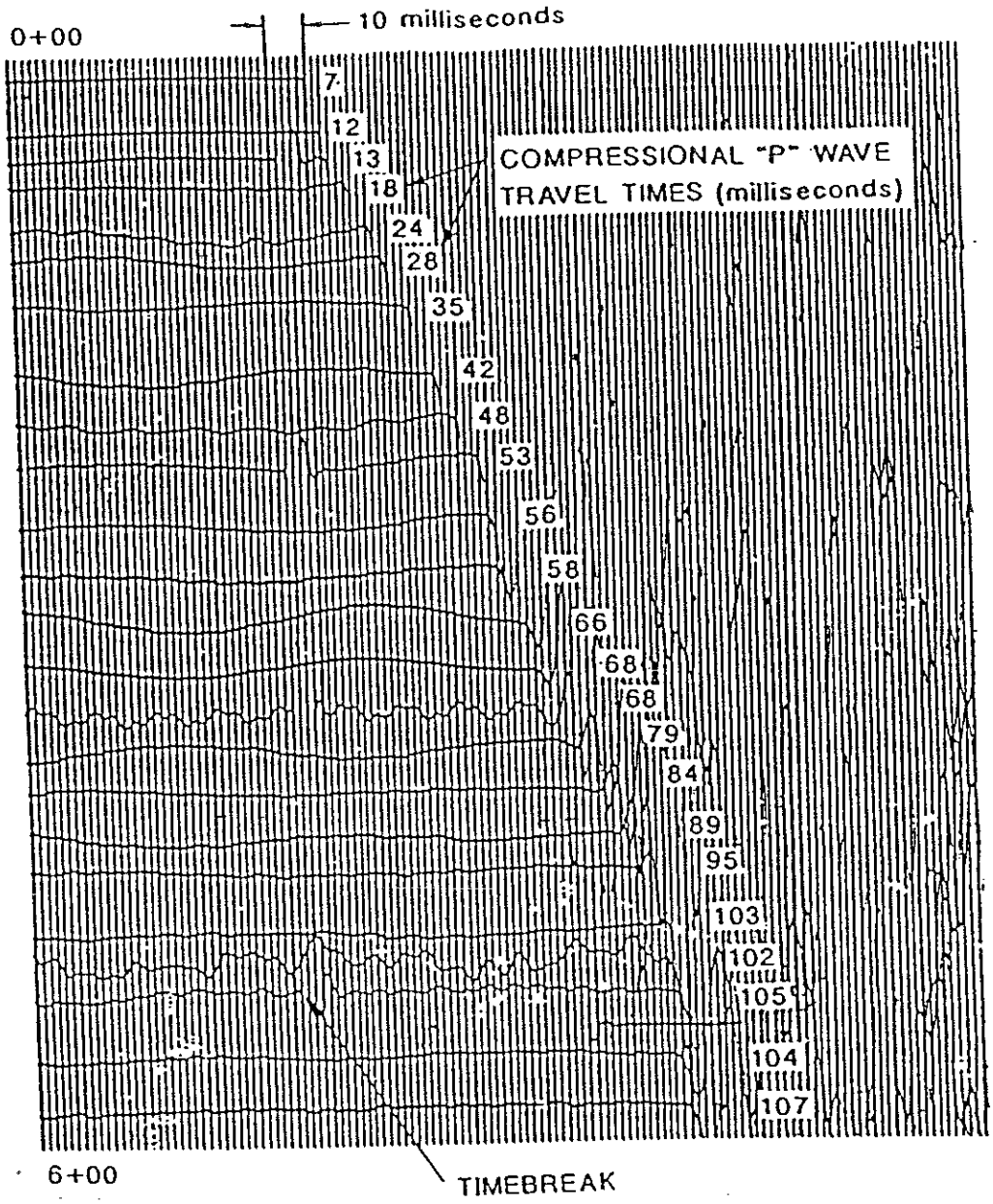
Explosive sources can be categorized as:

- Dynamite
- Primers
- Blasting agents

Choice of energy source is dependent on site conditions, depth of investigation, and seismic technique chosen as well as local restrictions. Explosive sources may be prohibited in urban areas where non-explosive sources can be routinely used. Deeper investigations usually require a larger energy source; therefore, explosives may be required for sufficient penetration.

## 5.4 Field Procedures

A single channel seismograph is the simplest seismic instrument and is used with a single geophone and usually a hammer source. The geophone is usually placed at a fixed location and the hammer struck at regularly increasing distances from the geophone. First wave arrival times are identified in the instrument display, logged in a field book, and immediately plotted on a time distance (T/D) plot. Single channel seismographs should only be used for the shallowest of surveys where the depth of investigation is less than 20 feet. A signal enhancement capability can somewhat expand the depth of investigation to 50 feet or more under ideal circumstances. Multichannel seismographs that increase the rate of data acquisition are more commonly used. With the appropriate energy source and geophone spacings, multichannel systems are used to record data from great depths, up to 1,000 feet and more.




24-TRACE SEISMIC REFRACTION ANOLOG RECORD. VERTICAL LINES ARE TIME LINES. NUMBERS ARE MEASUED FIRST-ARRIVAL TIMES FOR INDIVIDUAL GEOPHONES.

EXAMPLE SEISMIC REFRACTION ANALOG RECORD

The most commonly used method of seismic refraction surveying is reversed profiling. It is accomplished by setting out a straight-line array of geophones and then recording the signals caused by a source at one end and then the other, allowing the production of a two-dimensional subsurface cross-section.

At a minimum, data are recorded for each spread location twice. This is accomplished by recording with the energy source at each end of the spread. This procedure is termed reversing the profile. Reversed profiling results in measured travel times in two directions and is necessary to accurately map dipping interfaces and transition zones. Continuous profiling is accomplished by having the energy source location (shot point) of one spread coincident with an end or intermediate shot point of the succeeding spread.

Seismic refraction surveys may be conducted on a grid basis, or along a single line depending on the type of data required, site size, and the time and budget constraints. Setting out a grid of shotpoints allows a three-dimensional subsurface stratigraphic map to be produced. Additional seismic energy source points located along the profile will produce more seismic data with which to construct subsurface profiles and to control lateral variations in material and the resultant changes in seismic velocities. Additional survey techniques for assessing lateral variations include broadside shooting, in which the shotpoints and geophones are located along parallel lines, and fan shooting, in which the geophones are laid out in a fan shape with the shot point at its apex.

To acquire seismic refraction data, a specific number of geophones are spaced at regular intervals along a straight line on the ground surface; this line is commonly referred to as a seismic spread. The length of spread determines the depth of penetration; a longer spread is required for a greater depth of penetration. Spread length should be approximately three to five times the required depth of penetration. Required resolution will control the number of geophones in each spread and the distance between each geophone. Closer spacings and more geophones usually result in more detail and greater resolution.

Since the seismic method measures ground vibration, it is inherently sensitive to noise from a variety of sources such as traffic, wind, etc. Signal enhancement is a significant aid when working in noisy areas and with smaller energy sources. Enhancement capability is available in most single and multichannel systems. Enhancement is accomplished by adding a number of seismic signals from a repeated source. This causes the seismic signal to "grow" out of the noise level, permitting operation in noisier environments and at greater hammer-to-geophone spacings. The overall results provide a more accurate measurement of the first arrival time.

The locations of individual seismic spreads and profile lines should be consistent with the desired subsurface information. Where a bedrock depression of a feature is suspected, seismic lines should be oriented perpendicular to the suspected trend of the feature. Seismic cross profiles may be necessary to confirm depths to a particular refracting horizon, especially when there are steeply dipping layers involved as on the edge of the bedrock valley. At a site where little information is known about subsurface layering trends, at least two seismic lines oriented in "T" or "L" arrangement should be completed and the data assessed before further refraction profiling.

In addition to equipment and basic data considerations, other important presurvey concerns are:

- Topography
- Geology
- Vibration generating activities
- On-site utilities and other cultural features (buildings, etc.)

The topography of a site dictates whether or not surveyed elevations are required. If possible, refraction profile lines should be positioned along level topography. For highly variable topography, a continuous elevation profile may be required to ensure sufficiently accurate cross-sections and to permit the use of time corrections in the interpretation of the refraction data.

Knowledge of site geology can be used to determine the energy source. Some geologic materials, such as loose, unsaturated alluvium, do not transmit seismic energy as well and a larger energy seismic source may be required. Geologic conditions also dictate whether or not drilled shotholes are required.

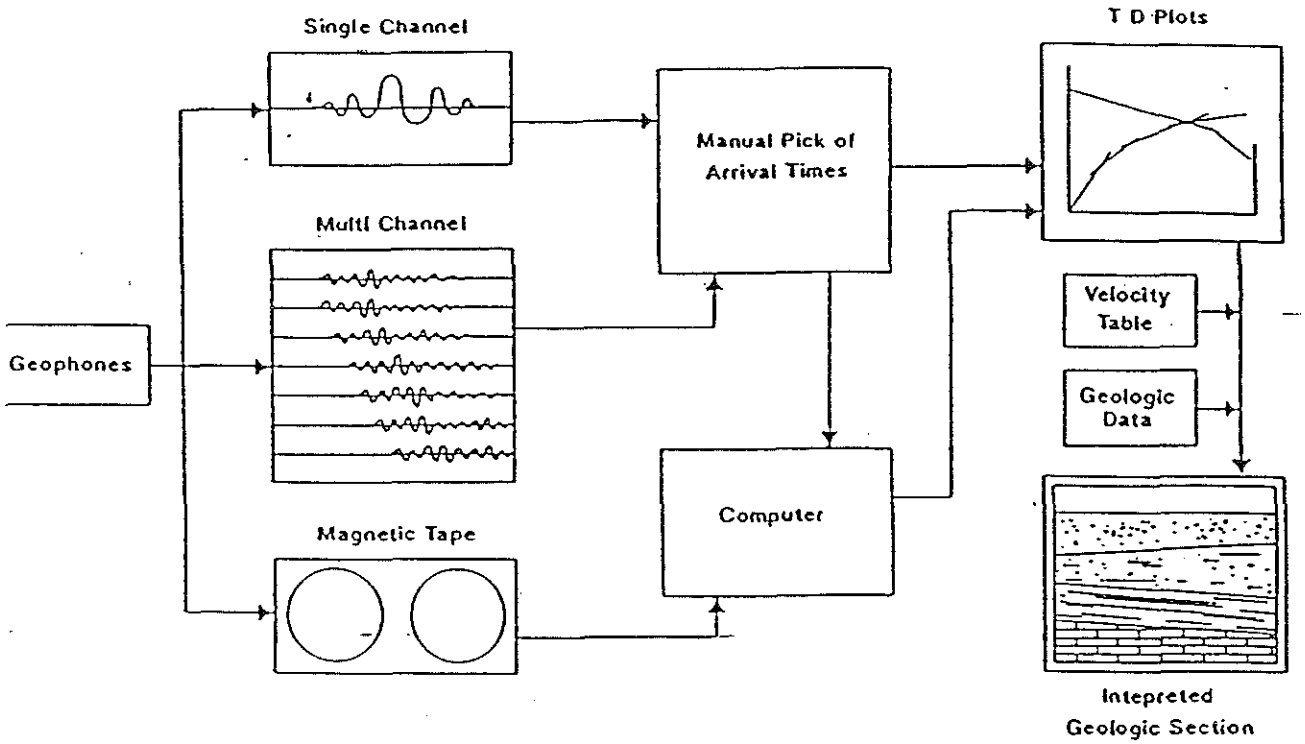
Cultural effects such as vibration generating activities, on-site utilities and buildings affect where data can be acquired, and where the lines are located. High volume traffic areas may require nighttime acquisition. If the survey is to be conducted near a building where vibration-sensitive manufacturing is conducted, data acquisition may be constrained to particular time intervals and appropriate energy sources must be used.

Data recorded in the field must include the coordinates (locations) of all receiver locations and shotpoints as well as specifics of the seismic energy source, electronic filtering and amplification used and in the case of direct read out seismographs, the travel times in milliseconds.

The data output of a seismograph depends upon the number of geophones used and the style of recording output. Multichannel seismographs may produce a travel-time versus distance chart which can be utilized directly to determine velocities and depths. Single channel recorders will require the operator to manually produce a travel-time chart of each shot as the source-receiver distance is changed. In all cases, the preferred format of data presentation is a graph in which travel time in milliseconds in plotted against source-receiver distance. From such a chart, the velocities of each layer can be obtained directly from the slope of each straight line segment, and information on layer parameters can be subsequently calculated. Figure 5 is a flow diagram showing the steps in processing and interpretation of seismic refraction data.

Seismic reflection surveys are generally used for deeper depths of investigation (hundreds of feet). Field procedures for seismic reflection are similar to those used in refraction. Reflection surveys are usually conducted with shorter receiver spreads and more geophones compared to a refraction survey for similar depths. In addition to the first arrival, numerous reflected arrivals are recorded at each geophone. Due to the many wave arrivals at each geophone and the degree of processing necessary to analyze these arrivals, most seismic reflection data are recorded digitally, and computer processing is required for detailed and refined interpretation.

Figure 5




Source: Benson, Glaccum, and Noel (1982)

FLOW DIAGRAM SHOWING STEPS IN PROCESSING AND INTERPRETATION OF SEISMIC REFRACTION DATA

The results of any seismic survey, refraction or reflection are usually presented in profile form showing elevations of seismic horizons. Data acquired on a grid basis can be contoured and used to construct isopach maps. Seismic velocities and, therefore, generalized material identifications should be presented on refraction profiles along with any test borings used for correlation to establish confidence in the overall subsurface data, both seismic and borings.

The success of any seismic survey is a direct function of the training and experience of the personnel involved. High quality field data must be obtained; poor quality data, instrumentation problems, etc. must be immediately recognized in the field and the appropriate measures taken to correct the situation. In the refraction interpretation process, the interpreter needs to be constantly aware of travel time anomalies, whether they be individual arrivals or along the entire spread. The interpreter should be aware of lateral velocity changes and apparent velocities, and be capable of calculating time velocities, dip angles, etc. The text book situation of a flat lying, two or three layer case, is the exception. Reflection also requires highly trained field personnel and interpreters knowledgeable of the sophisticated computer processing techniques.

## 5.5 Interpretation

### 5.5.1 Refraction Data Interpretation

Interpretation of seismic refraction data involves solving a number of mathematical equations with the refraction data as it is presented on a travel-time versus distance chart. Analog seismic refraction data can be processed by plotting the data by hand and using a hand calculator. Travel times for the first arrival waves at each geophone are measured from the analog record. For a site containing horizontal stratigraphic layers of increasing velocity, the travel time chart will consist of a series of overlapping straight line segments of decreasing slope. Each line segment (1/(time/distance)) is equal to the seismic velocity in a layer. Using these velocities the critical angle of refraction for each boundary can be calculated using Snell's Law. Then, utilizing these velocities, and angles and the recorded distances to crossover points (where line segments cross), the depths and thicknesses of each layer can be calculated using simple geometric relationships.

Thicknesses of velocity layers are calculated by either the critical distance or time intercept methods (Redpath, 1973). Accurate depth calculations are dependent on the assumption that the velocity of each geologic layer increases with depth. If that is not the case, additional corrections must be applied.

Critical Distance Method. The critical distance $X_c$ is determined by constructing a line from the intersection of the two straight-line velocity segments perpendicular to the X-axis. Depths to refracting horizons are calculated by using the critical distance and the layer velocities.

Time Intercept Method. Time intercept values for each layer are determined by extending the velocity line segments to intersect the y-axis. That intersection is the time intercept for that layer. Depths using the time intercept method are calculated from the intercept time and the layer velocities.

The section developed in Figure 5 was produced using the critical distance method. If the profile had not been "reversed," that is, had there not been a shot at each end, the dipping interfaces and the detail would not have been resolved. Important corrections which should also be evaluated are:

- Depth of shot
- Topography
- Velocity inversions

There are a number of complicating factors. Where reverse profiles indicate dipping boundaries, calculation of dips, true depths and true velocities involve more complicated equations. Furthermore, corrections for differing elevations and varying thicknesses of weathered zones must often be made. Fracturing and weathering generally reduce seismic velocity values in bedrock. Consequently, travel-time plots with late arrivals must be evaluated carefully to determine if the late arrival times (slower velocities) are due to overburden conditions or fractured/weathered bedrock.

Very thin layers or low velocity zones often complicate the travel-time chart as well. Although not the usual case, one constraint on refraction theory is that material velocities ideally should increase with depth. If a velocity inversion exists, i.e. where a low velocity layer is overlain by a higher velocity layer, depths and seismic velocities can be calculated, but the uncertainty in calculations is increased unless borehole velocity data are available. Irregular boundaries cannot be adequately resolved with time-distance analysis. Instead, another form of analysis involving delay-time is used in these situations.

Although seismic refraction is very useful in confirming subsurface structures and performing reconnaissance surveys, it should be noted that multiple interpretations for each data set are possible. Additional independent information for correlation purposes is very important to the interpretation.

### 5.5.2 Reflection Data Interpretation

For smaller depths of investigation, reflection surveys are usually conducted with shorter spreads but with more geophones compared with a refraction survey. In addition to the first arrival, the entire waveform is of interest in seismic reflection. Due to the arrival of many reflected waves at each geophone and the large extent of processing necessary to establish coherency for these arrivals, most seismic reflection data are recorded digitally, and computer processing is required for detailed and refined interpretation. Corrections that should be applied include, but are not limited to:

- Normal move-out (correction for source-to-geophone distances)
- Thickness overburden and layers
- Migration of reflector points
- Signal filtering and enhancement

After computer processing, the data are printed as a variable density plot, on which waveforms show discrete reflectors which represent material boundaries. A cross-section based on horizontal distance versus travel time can be constructed from this plot. Only after a depth calibration is provided by means of drilling or velocities determined by uphole/downhole or refraction surveys can a cross-section be drawn.

## 5.6 Advantages and Disadvantages

The seismic refraction technique, when properly employed, is the most accurate of the geophysical methods for determining subsurface layering and materials. It is extremely effective in that as much as 2,000 lineal feet or more of profiling can be acquired in a field day. The resulting profiles can be used to minimize drilling and place drilling at locations where borehole information will be maximized resulting in cost effective exploration. A standard drilling program runs the risk of missing key locations due to drillhole spacings. This risk is substantially reduced when refraction is used which produces a continuous subsurface profile.

In summary, the advantages and limitations of the seismic techniques are:

**Seismic Refraction**

Advantages

- Material identification
- Subsurface data over broader areas at less cost than drilling
- Relatively accurate depth determination
- Correlation between drillholes
- Preliminary results available almost immediately
- Rapid data processing

Limitations

- As depth of interest and geophone spacings increase, resolution decreases
- Thin layers may be undetected
- Velocity inversions may add uncertainty to calculations
- Susceptible to noise interference in urban areas which require use of grounded cables and equipment, signal enhancement and alternative energy sources

**Seismic Reflection**

Advantages

- Higher resolution
- Velocity inversions do not affect accuracy
- Smaller energy sources required
- Shorter spreads necessary

Limitations

- Precision interpretation requires computer processing
- Generally more expensive than refraction
- Not fully proven for shallow applications

## 6.0 QUALITY ASSURANCE RECORDS

Field data will be recorded in Field Logbooks or data logging sheets recording directly from the data logger. All data entries will have the following information: date, site name, start and end times (in military time), name of personnel on site, and weather.

## 7.0 REFERENCES

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# F706
# *RESISTIVITY METHOD*

# GEOPHYSICS: RESISTIVITY METHOD

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information for using and interpreting electrical resistivity surveying.

## 2.0 SCOPE

This SOP indicates the normal methods of interpreting and implementing resistivity data. The description of field implementation methods, data acquisition and recording are described herein.

## 3.0 DEFINITIONS

Finite-element modeling - A numerical method of approximating a solution to differential equations.

Hertz - A unit of frequency. One hertz equals one cycle per second.

Impedance - The apparent resistance to the flow of alternating current; analogous to resistance in a direct current circuit.

Milliamp - A unit of electric current flow, equal to one thousandth of an ampere.

Sinusoidal - An adjective describing a curve in the pattern of a sine function (mathematics).

Sounding - Measurement of geoelectrical properties as they vary with depth.

Square wave - A waveform consisting of alternating positive and negative portions.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that the project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for ensuring that the personnel operating and interpreting the geophysical data are trained, skilled in that endeavor, so far as to receiving documentation on the training and experience of the operating personnel.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the geophysical technique and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the activities have been briefed and trained to execute these procedures.

## 5.0 PROCEDURES

### 5.1 Overview

Electrical resistivity surveying is an active geophysical technique that utilizes electrical measurements obtained on the ground surface to determine physical properties of subsurface materials. Typically an electric current is applied to the earth using two electrodes (current electrodes $C_1$ and $C_2$); the resulting potential difference or voltage is measured between a second pair of electrodes (potential electrodes $P_1$ and $P_2$) as shown on Figure 1. An "apparent resistivity" is then calculated using values for the applied current, measured voltage, and electrode separation.

The values measured during a field program are known as "apparent" resistivities because they can be a composite of resistivity values for several layers. For a single isotropic homogeneous material, the apparent resistivity would equal the true resistivity.

Resistivity and its reciprocal, electrical conductivity (1/resistivity = conductivity), are inherent properties of soil, rock, and groundwater. The resistivity of an earth material depends on the following:

- Material composition
- Water content (porosity and degree of saturation)
- Salinity or ion content of the water
- Permeability
- Temperature

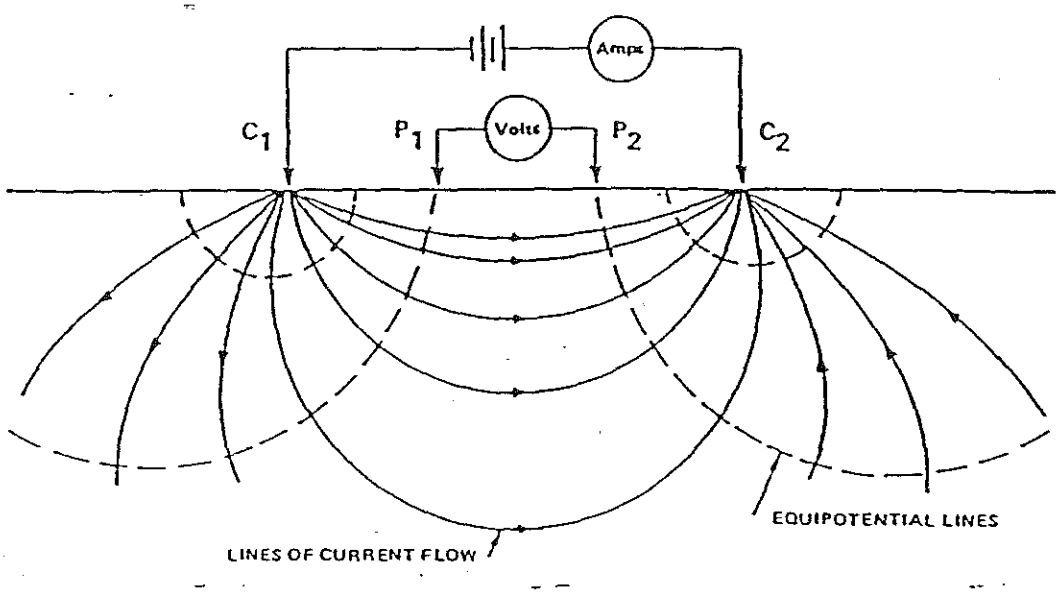
Most soil and rock materials are relatively poor electrical conductors (i.e., exhibit high resistivities) compared with groundwater. An applied electrical current is conducted almost entirely by water in the pore spaces or fractures of soil or rock rather than by the soil or rock alone. This applies to the unsaturated and vadose zones, because in general there is some moisture in unsaturated media. Pure water is non-conductive, but most groundwater contains dissolved salts and hence is somewhat conductive.

The approximate ranges of resistivity for common soil and rock types are shown on Figure 2. The ranges of resistivity values for a single material generally indicate resistivity variations between dry and water-saturated conditions. Dry sands, gravels, and massive unweathered rock typically exhibit relatively high resistivities; whereas, clays, clayey tills, water-saturated sediments, and weathered rock (chemically broken down to clays) tend to have lower resistivities.

Resistivity measurements are commonly used to delineate either changes in resistivity with depth or lateral variations in resistivity. These applications are known respectively as:

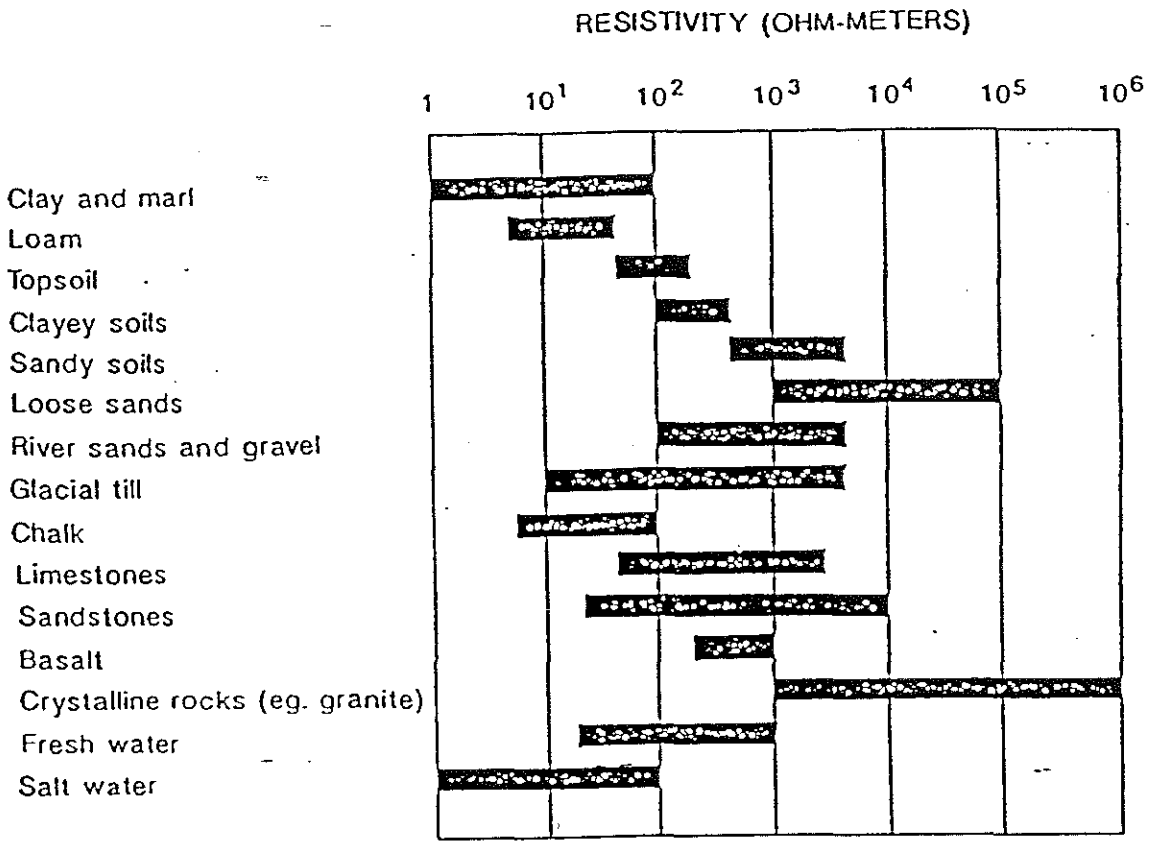
- Vertical electrical soundings (VES)
- Horizontal profiling

FIGURE 1




Electric current is applied to the earth between current electrodes, $C_1$ and $C_2$. Potential field generated by the current is measured between potential electrodes, $P_1$ and $P_2$. Source: U.S. Army Corps of Engineers, 1979.

BASIC CONFIGURATION OF AN ELECTRICAL RESISTIVITY SURVEY




Ranges of values reflect influence of variations in porosity, saturation, and ground-water conductivity.

Source: Benson, Glaccum, and Noel, 1983.

TYPICAL RESISTIVITIES FOR COMMON ROCKS AND SOILS

In VES surveys the center of an electrode array is kept at a fixed position, and resistivity measurements are obtained at successively larger electrode spacings. Measurements at the longer electrode separations sense deeper into the earth to identify geoelectrical layering in soil and rock. These data are often used to identify the water table, clay layers, and the bedrock surface, and to select optimum electrode spacings for horizontal profiling surveys

In horizontal profiling, the electrode spacing is kept constant and the array is moved across the survey area. Profiling measurements are often repeated with at least two different electrode separations to identify lateral variations at more than one depth.

A variety of electrode arrays are used for resistivity surveys. The most common ones will be discussed in this section. In all of the arrays listed below, the electrodes are arranged in a straight line. Differences between the arrays consist of variations in electrode spacing and relative position. The most commonly used electrode arrays (see Figure 3) are:

- Wenner (and Lee modification of Wenner)
- Schlumberger
- Dipole-dipole

These three arrays measure the induced electrical (potential) field differently, thus the arrays have different applications. The Wenner array measures an "average" of the induced potential because its potential electrodes are relatively far apart. This averaged measurement renders the Wenner array suitable for VES surveys, but it is less sensitive to horizontal variations in resistivity.

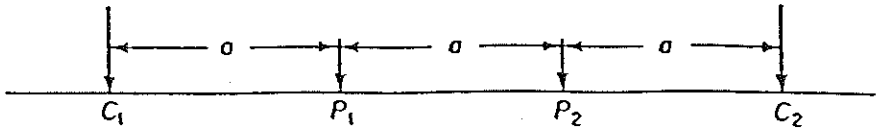
The Lee modification of the Wenner array involves the addition of a third potential electrode halfway between $P_1$ and $P_2$ (see Figure 3). Three potential measurements are taken, using electrodes $P_1 - P_2$ (normal), $P_1 - P_0$ (Lee left), and $P_2 - P_0$ (Lee right). Apparent resistivities are calculated for the Lee left and right measurements. If the left and right measurements do not each equal one-half of the normal measurement, there is either a measurement error or a lateral variation in resistivity in the vicinity of the potential electrodes.

In the Schlumberger array the potential electrodes are relatively close together compared to the current electrodes, and the array measures the first derivative of the induced potential field. The Schlumberger array's performance is comparable to that of the Wenner array for VES applications, but has greater sensitivity in horizontal profiling. Generally, only the current electrodes are moved in a Schlumberger VES survey. This is simpler than Wenner VES measurements, in which four electrodes must be moved, but it can result in larger measurement errors. Because of the closely-spaced potential electrodes the voltages measured in a Schlumberger survey are smaller than those measured in a Wenner survey, and an error in potential measurement has a greater effect on Schlumberger-calculated resistivities. At sites where many local variations in resistivity occur near the ground surface, Schlumberger results may be less noisy because the potential electrodes are kept in the same material for several readings. Wenner results for the same area will be more noisy because the potential electrodes are in different media for each reading.

Ia WENNER

$\rho_a = 2\pi a \Delta V / I$

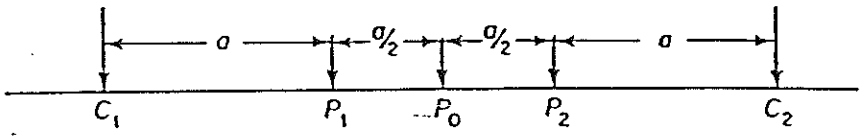
$\Delta V$ taken between $P_1 P_2$




Ib LEE MODIFICATION OF WENNER

$\rho_a = 4\pi a \Delta V / I$

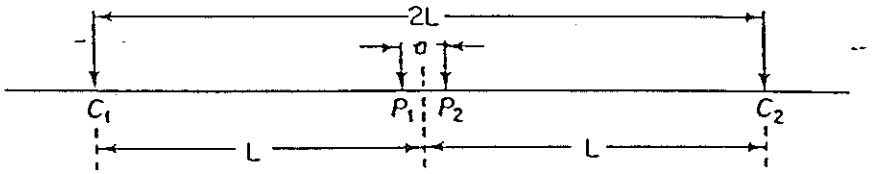
$\Delta V$ taken between $P_1 P_0$ and $P_0 P_2$




II SCHLUMBERGER

$\rho_a = \frac{\pi L^2}{a} \frac{\Delta V}{I}$

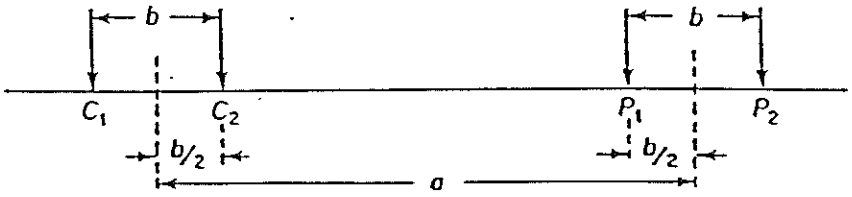
$\Delta V$ taken between $P_1 P_2$




III OIPOLE-OIPOLE

$\rho_a = \pi (a^3/b^2 - a) \Delta V / I$

$\Delta V$ taken between $P_1 P_2$




ELECTRODE ARRAYS USED IN SOUNDING AND PROFILING SURVEYS

The Wenner and Schlumberger arrays have some similarities. Both can be used for profiling or VES surveys, and both have a maximum depth of investigation related to the current electrode separation (approximately one-third of the Wenner "a-spacing" or one-ninth of the Schlumberger current electrode separation). Schlumberger profiling measurements have greater sensitivity to lateral resistivity changes, and Schlumberger VES data have less error due to heterogeneous near-surface materials. Wenner measurements are more accurate at sites where small potential voltages are induced, including sites with low resistivity (high conductivity) clays. Either Wenner or Schlumberger arrays may be used for reconnaissance and detailed measurements by varying the spacing between profile traverses or sounding locations. Widely spaced traverses or soundings are used for reconnaissance surveys or for delineation of large targets (horizontally extensive clay or gravel layers). Closely spaced data are required for identification of localized features such as discrete zones of leachate migration, etc.

Dipole-dipole measurements represent the second derivative of the induced potential field; they are thus more sensitive to lateral changes in resistivity than the Wenner or Schlumberger arrays, but less sensitive to changes with depth. A dipole-dipole survey is better-suited to locating discrete features (buried metal, igneous dikes, solution cavities), and should not be used for identification of soil and rock layering.

## 5.2 Applications

Contrasts in resistivity for some geologic materials (see Figure 2) make resistivity surveying a valuable technique for many applications including:

- Depth to groundwater
- Delineation of conductive contaminant plumes and/or buried wastes
- Locating fresh/salt water interfaces
- Detecting a perched water table
- Distinguishing bedrock and sediment lithologic types and contacts
- Identification of zones of weathered bedrock, fractures, and possibly solution cavities

Examples of resistivity applications including identification of buried stream channels, mapping the groundwater table, and clay layers may be found in Zhody et al. (1974) and Yazicigil and Sendlein (1982).

Resistivity surveys have occasionally been applied to the problem of detecting electrically resistive contaminants; however, this procedure is generally not possible. Successful applications of resistivity in organic contaminant identification requires (1) conductive contaminants (landfill leachate, chlorides, iron oxides, dissolved nitrates and salts) associated with the organic compounds, or (2) a layer of organic product (hydrocarbons including gasoline, PCBs) several feet thick which displaces the groundwater table.

Resistivity data are well-suited for correlation and verification by a variety of geophysical techniques and/or test borings. Possible correlative geophysical techniques for the applications listed above include seismic retraction, electromagnetic terrain conductivity, ground penetrating radar (GPR), and magnetometry.

## 5.3 Equipment

Basic field equipment needed for resistivity surveying includes:

- Two current electrodes
- Two or three potential electrodes
- Insulated connecting cables
- Non-conductive fiberglass measuring tapes
- Source of electric current
- Voltage measurement device

Resistivity instrumentation comes in a variety of designs with widely varying capabilities. Advantages and limitations of four popular designs will be discussed in this section.

The simplest resistivity instruments are known as "DC" (direct current) devices. They apply a direct current to the earth and measure the resulting DC potential with a high-impedance (at least $1 \times 10^6$ ohm) volt meter. Because SP (self-potential) voltages can adversely affect the accuracy of simple DC resistivity measurements, these instruments usually contain a "nulling" or "balancing" circuit to remove the SP effect; although SP constitutes a form of noise in a resistivity survey, SP measurements can also be used as a geophysical exploration technique (see Section 8.50). If SP is varying rapidly in the area of investigation, then its effect is nearly impossible to remove or compensate; for this reason, simple DC resistivity measurements are not suitable for all field areas (see Section 8.5.1 for a discussion of SP sources).

A more versatile resistivity meter is known as the low-frequency "AC" (alternating current) type. This instrument uses a sinusoidal applied current, usually of only a few hertz, to avoid some of the interference caused by SP. Both the DC and low-frequency AC meters are best-suited for relatively shallow investigations, with depths of investigation less than about 100 feet, in soils that are neither highly conductive nor highly resistive. These limitations are imposed by the small battery-powered current transmitters used. Current output of these units is measured in tens of milliamps at less than one thousand volts.

More powerful resistivity equipment is also available, using sinusoidal AC or square-wave DC transmitters powered by portable electric generators. These units often have a transmitter and receiver mounted in separate housings to provide greater versatility and to minimize electrical interference between the transmitting and receiving circuits. They also have the capability of producing up to tens of amperes of current at several thousand volts, sufficient for surveys in highly resistive or conductive media at maximum depths much greater than battery powered instruments.

Recent innovations in electronics design have resulted in a fourth type of resistivity meter that fills a niche between the standard battery and generator powered AC instruments. These units are also battery-powered, but produce electric currents with unique waveforms. Voltage measuring circuits in these devices are designed to recognize the specific waveform produced by the transmitter, thus enabling measurement of weak potentials in somewhat noisy conditions. Signal enhancement (summing of a few voltage measurements) is usually offered with these instruments and also contributes to improved resolution. This type of resistivity meter is capable of operation in more resistive or conductive media than the low-frequency AC meters, and can also be used to investigate deeper structures.

## 5.4 Field Procedures

Field procedures involve placing electrodes at the intended separations, connecting the electrodes to the transmitter and receiver, and obtaining current and potential measurements. Electrode locations should be determined with non-conductive measuring tapes to avoid providing an alternative path for the applied current. Fiberglass tapes are commonly used. Most resistivity surveys are performed with metal electrodes which are driven into the ground. Steel and copper-clad steel are common electrode materials, although other metals may be used. Electrodes of dissimilar metals should not be used during a survey (e.g. three steel electrodes and one aluminum electrode) because unusually large self-potentials can be generated. Electrodes are usually driven one to three feet into the ground. Water is poured around each electrode, if needed, to decrease the resistance between the electrode and earth materials. Copper sulfate solutions have historically been used to improve electrode contact, but tap water is usually sufficient.

At sites with strong self-potential noise effects, use of DC resistivity instrumentation may necessitate non-polarizing electrodes. These special electrodes are commonly of the porous-pot type, consisting of an unglazed ceramic pot containing a metal electrode and a saturated electrolytic solution. The solution must be of a salt of the same metal as the central electrode; e.g., a solution of copper sulfate is often used with a copper electrode. The porous pot is placed on the ground surface, and electrical contact with the earth is achieved by seepage of the electrolytic solution through the porous ceramic. Finally, the electrode is connected to the resistivity instrumentation by insulated wire.

Quality assurance is important in resistivity field procedures. This entails careful measurement of electrode positions, checking resistances across potential and current electrodes to ensure good contact with the earth, and plotting calculated apparent resistivities in the field to quickly make identification of spurious readings. Instrument calibration is not usually of concern because the equipment is calibrated by its manufacturer. Verification of the equipment's operating condition can be obtained by repeating resistivity measurements at a known location prior to conducting field work.

Careful planning is another significant factor in conducting resistivity surveys. Parallel lines should be tied together with a perpendicular line, or by using another geophysical technique such as EM or seismic. To minimize errors from fences or other cultural features, electrode arrays should be placed perpendicular to metal fences or other linear conductive objects. Topographic effects are minimized if the electrodes are maintained at nearly the same elevation; a VES survey is best performed along hillside contours, not up and down the hill.

## 5.5 Interpretation

### 5.5.1 Data Analysis

Analysis of resistivity data involves different procedures for horizontal profiling, VES surveys, and dipole-dipole surveys. Horizontal profiling data is contoured or plotted on linear graph paper, with apparent resistivity values on the y-axis and distance along the traverse on the x-axis. The contour map or profiles are then examined for relative variations in resistivity which may be indicative of the intended target body. An example of Wenner profiling data is provided on Figure 4.

Wenner and Schlumberger VES data are plotted on log-log graph paper with apparent resistivity values on the y-axis and Wenner a-spacings or the Schlumberger current electrode separations on the x-axis (Figure 5). Until a few years ago, interpretation would have next been accomplished by comparison of the field data with published master curves. Examples of the curves and their use may be found in Orellana and Mooney (1966), Keller and Frischknecht (1966), Dobrin (1976), Telford et al. (1976), Van Nostrand and Cook (1966), and Zhody et al. (1974). This technique is slow, inaccurate, and limited in application because curves are available only for two and three layer cases at a few resistivity contrasts.

Currently, VES interpretations are performed using a computer and the linear filter algorithm described by Ghosh (1971a and 1971b) and Koefoed (1979). This algorithm operates very quickly on any computer, from a mainframe to a Laptop, and provides greater accuracy and versatility than is possible with curve matching techniques. A contractor should clearly identify the modeling technique used in the interpretation.

Computer-aided modeling can be performed in two manners. Forward modeling entails computation of theoretical resistivity values from a layer thickness/resistivity model supplied by the interpreter. Agreement between the field and theoretical curves in the model is obtained by subsequent trial and error refinement of the layer parameters (thicknesses and resistivities). Boring or test pit logs, if available, should be used to confirm the resistivity modeling results.

Inverse modeling also begins with computation of theoretical resistivity values from layer parameters supplied by the interpreter, but refinement of the layer parameters is automatically performed by the computer code. The final product of an inverse modeling session is a set of layer parameters and a corresponding theoretical curve which provide the best possible fit to the field data. This "numerically correct" interpretation must be examined by a geologist or geophysicist to ensure that the model is geologically reasonable. Again, actual field data from a boring or test pit should be used to check the model.

Dipole-dipole resistivity analysis is considerably different from horizontal profiling or VES analysis. Dipole-dipole data are displayed in a two-dimensional pseudosection format (Figure 6), and the analysis is thus performed by two-dimensional numerical modeling. An example of finite-element modeling of dipole-dipole data may be found in Rijo (1977). Note that the complexity of this finite-element modeling requires a well-trained interpreter and a mini-computer; these restrictions will limit the number of contractors qualified to perform dipole-dipole interpretation.

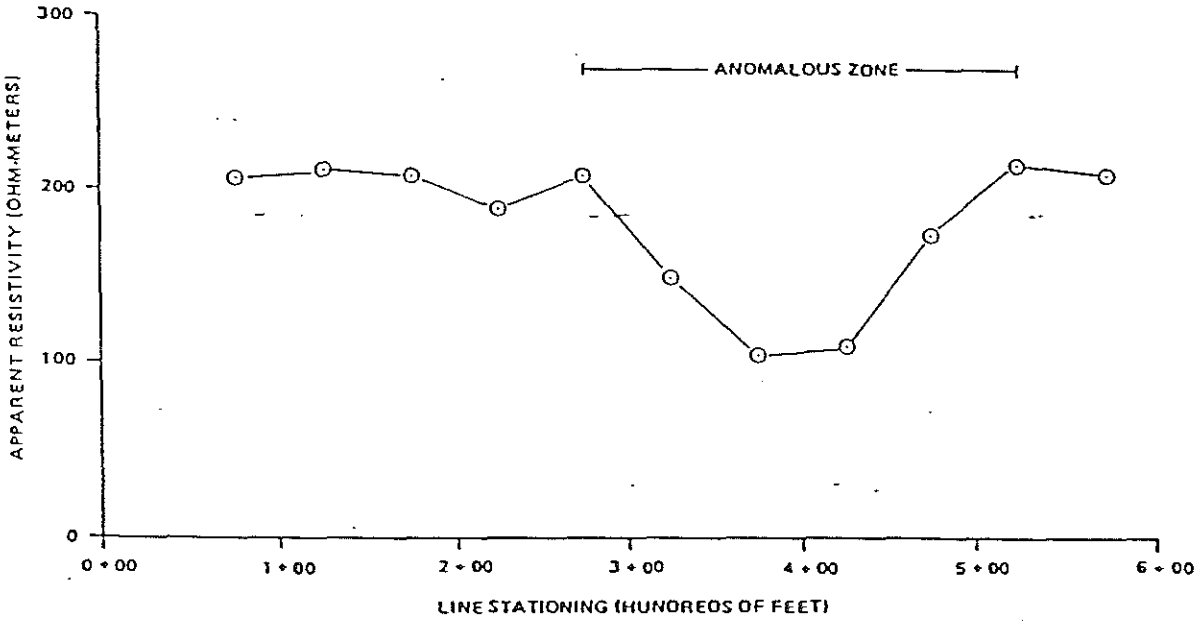
FIGURE 4



A.) DATA:

| CURRENT ELECTRODE LOCATIONS (50 FOOT "A" SPACING) | CENTER OF ARRAY | APPARENT RESISTIVITY (OHM-FEET) |
|---|---|---|
| 0 + 00 — 1 + 50 | 0 + 75 | 672 |
| 0 + 50 — 2 + 00 | 1 + 25 | 692 |
| 1 + 00 — 2 + 50 | 1 + 75 | 679 |
| 1 + 50 — 3 + 00 | 2 + 25 | 613 |
| 2 + 00 — 3 + 50 | 2 + 75 | 672 |
| 2 + 50 — 4 + 00 | 3 + 25 | 482 |
| 3 + 00 — 4 + 50 | 3 + 75 | 341 |
| 3 + 50 — 5 + 00 | 4 + 25 | 351 |
| 4 + 00 — 5 + 50 | 4 + 75 | 567 |
| 4 + 50 — 6 + 00 | 5 + 25 | 695 |
| 5 + 00 — 6 + 50 | 5 + 75 | 679 |

B.) INTERPRETATION:



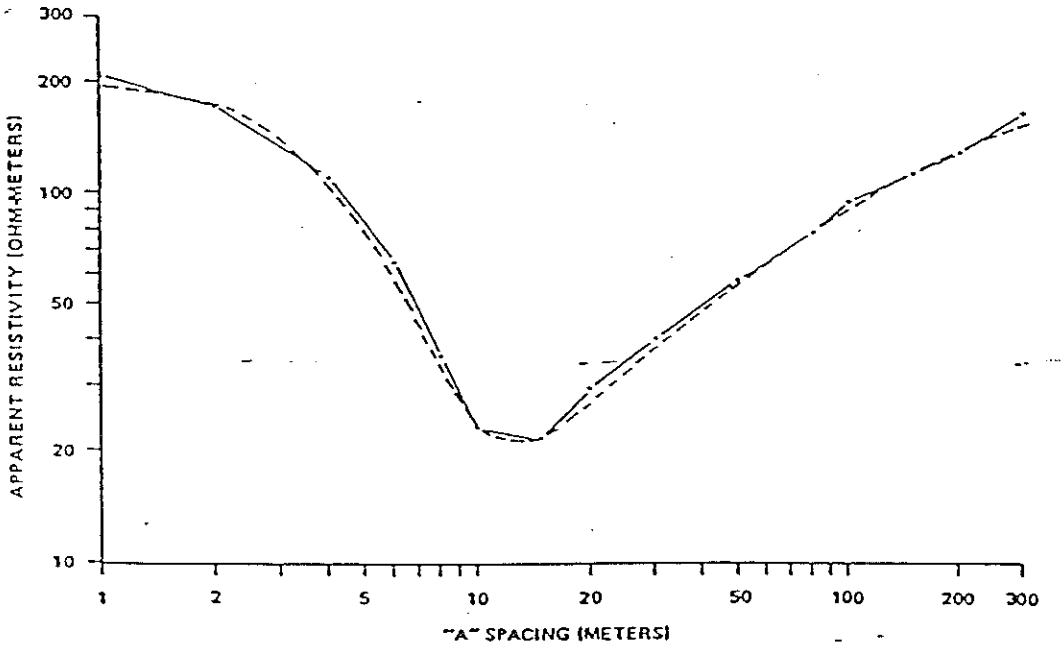

EXAMPLE OF WENNER PROFILING RESISTIVITY DATA (A) AND INTERPRETATION (B)

FIGURE 6

A.) DATA:

| "A" SPACING (METERS) | APPARENT RESISTIVITY (OHM-METERS) |
|---|---|
| 1 | 205 |
| 2 | 172 |
| 4 | 110 |
| 6 | 64 |
| 8 | 36 |
| 10 | 23 |
| 15 | 22 |
| 20 | 30 |
| 30 | 40 |
| 60 | 69 |
| 80 | 79 |
| 100 | 95 |
| 150 | 111 |
| 200 | 130 |
| 300 | 165 |

B.) INTERPRETATION:




| | | 1st LAYER | 2nd LAYER | 3rd LAYER |
|---|---|---|---|---|
| THEORETICAL MODEL | RESISTIVITY RATIOS | 1 | 0.05 | 1 |
| | THICKNESS RATIOS | 1 | 3 | |
| FIELD CURVE | | | | |

INTERPRETATION

| | THICKNESS | RESISTIVITY |
|---|---|---|
| 1st LAYER | 3 METERS | 200 OHM-METERS |
| 2nd LAYER | 9 METERS | 10 OHM-METERS |
| 3rd LAYER | ——— | 200 OHM-METERS |

EXAMPLE OF WENNER VES DATA (A) AND INTERPRETATION (B)

### 5.5.2 Presentation of Results

Horizontal profiling data are contoured or presented as linear-linear plots of apparent resistivity versus distance along a traverse. An example of the profiling plotting technique is provided on Figure 4.

VES data are plotted on log-log graphs with apparent resistivity values on the y-axis and Wenner a-spacings or the Schlumberger current electrode separations on the x-axis. See Figure 5 for an example. Layer parameters used in VES modeling, i.e. layer thicknesses and resistivities, must be included with each VES plot, as shown on Figure 5.

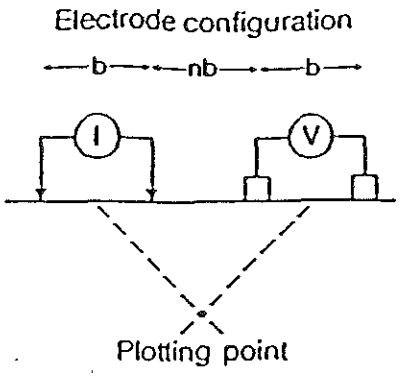
Dipole-dipole data are shown as pseudosection plots, usually with resistivity values contoured (see Figure 6). A cross section of the inferred geologic model should accompany the pseudosection plot.

### 5.3.3 Interpretation of Results

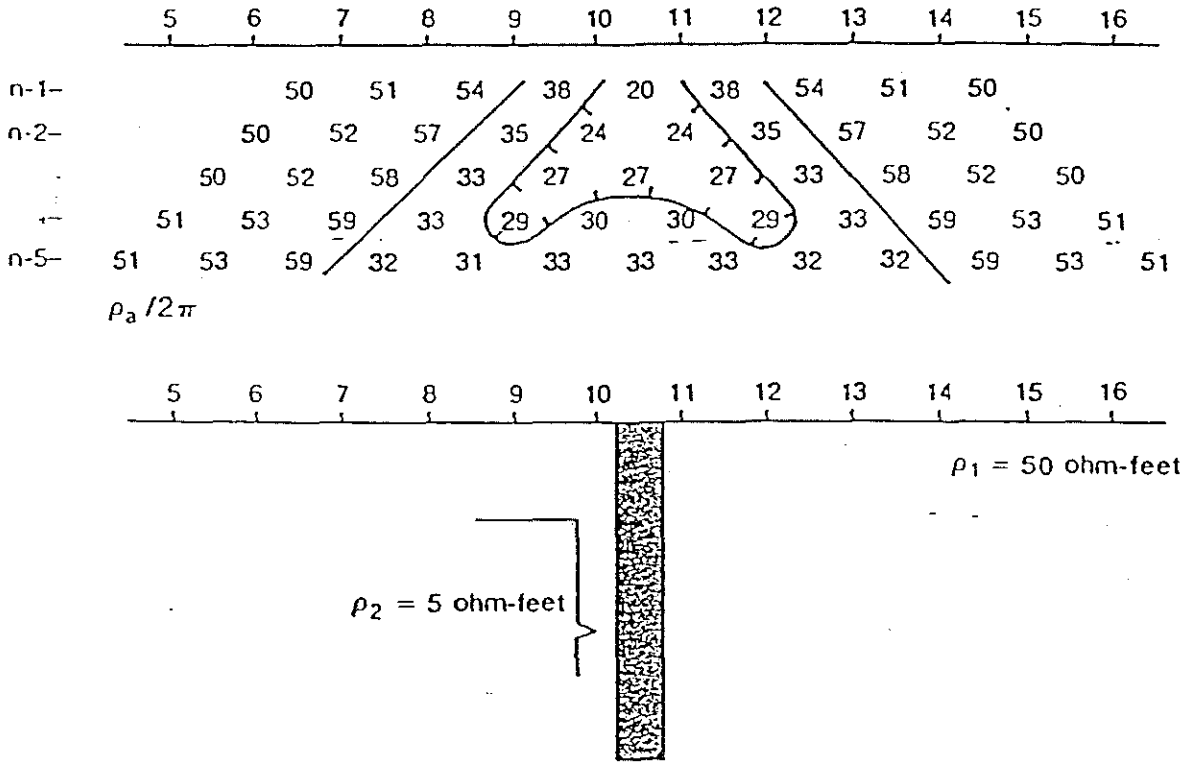
Interpretation of resistivity data entails comparing resultant ranges of resistivity values with natural earth materials or manmade objects likely to be present. Horizontal profiling data and VES modelling results are directly indicative of the resistivities of the materials encountered: higher resistivity values represent more electrically resistive materials (such as sands or gravels). Figure 4 shows Wenner profiling data over a localized zone of anomalously low resistivity soil. Figure 5 shows Wenner resistivity sounding data over a clay seam that occurs between more resistive soil layers.

Dipole-dipole interpretation is more subjective, and requires an experienced interpreter. The complexity of dipole-dipole interpretation arises from the lack of correspondence between a dipole-dipole pseudosection and the actual resistivities of the earth materials investigated. As a simple example, a low-resistivity vertical dike will produce a dipole-dipole anomaly in the shape of an inverted letter "V". Although the dike has low resistivity, the anomaly will contain both low and high resistivity values which could be misinterpreted by inexperienced personnel.

Correlation of resistivity data with other geophysical data sets, borehole geologic logs, or borehole cores and samples, is necessary to more accurately identify the materials or structures inferred from the resistivity results. Estimates of layering thicknesses from resistivity modeling typically have to be compared with seismic refraction or geologic data because of the imprecision inherent in resistivity layer calculations. The imprecision is caused by the non-uniqueness of resistivity data: many different models can produce theoretical curves which nearly fit the field data. A knowledgeable interpreter is thus needed to successfully integrate the resistivity results with other data, including geologic information regarding the site of interest.




PSEUDOSECTION PLOTTING FORMAT




EXAMPLE PSEUDOSECTION AND GEOLOGIC MODEL

EXAMPLE OF DIPOLE-DIPOLE RESISTIVITY PSEUDOSECTION FORMAT

## 5.6 Advantages and Disadvantages

Some of the advantages of resistivity surveying include the general portability of the equipment, the potential for in-field data reduction by using portable computers with horizontal profiling and VES surveys, and the generation of information which cannot be obtained with other methods at a similar cost. Equally important are the limitations inherent in resistivity surveying, which will be reviewed below. Knowledge of these limitations is critical to avoid misapplication of the resistivity technique.

First, the resistivity surveying methods can be carried out only in media which are neither extraordinarily conductive or resistive. If electrodes are placed in very conductive material, e.g., a clay layer, then the applied current flow is trapped in the conductive layer. A bedrock layer underlying the clay, could remain undetected because virtually none of the current would pass through the rock.

In very resistive materials, such as talus, resistivity surveying often cannot be performed because poor electrode contact prevents introduction of current into the earth. Marginal cases may be aided by wetting the electrodes to decrease earth resistance, but in severe cases the resistivity method must be replaced with another technique.

Another limitation is the size of a target body which can be detected by resistivity surveys. Thin layers, or targets of limited lateral extent, may be undetectable because the measured potentials integrate the effects of a large volume of material. This difficulty can be reduced if the minimum size and resistivity contrast of the expected target is known before the field measurements are begun. Numerical modelling can then be performed to select the most effective electrode array and spacing to identify the desired target. This approach is particularly effective in planning VES and dipole-dipole surveys.

Cultural interference is another serious limitation of resistivity surveying. Interference from metal fences can be minimized by orienting electrode arrays perpendicular to the fence. The same approach can be used with underground utilities, but in general an area of utilities is best avoided altogether.

All reports must include a statement of the field and computer methods used, including calibrations and correlations with other geologic or geophysical data.

## 6.0 QUALITY ASSURANCE RECORDS

All data will be recorded in Field Logbooks and/or data logging sheets designed for this procedure. All data will be entered with the following basic information: date, start and end times (military time), location, personnel on site, and weather.

## 7.0 REFERENCES

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# F707
# *GEOPHYSICS: SELF-POTENTIAL*

# GEOPHYSICS: SELF-POTENTIAL

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information for using self-potential geophysical methods.

## 2.0 SCOPE

This SOP provides a description of techniques, field applications, interpretation and equipment used in the application of self-potential methods.

## 3.0 DEFINITIONS

Bioelectric activity - Electric phenomena generated by vegetation.

High-impedance millivolt meter - An instrument capable of measuring small voltages without drawing excessive electric current.

Non-grounded case - An instrument case that is not electrically in contact with either the earth or the instrumentation housed in the case.

Non-polarizing electrodes - Electrodes which are free of potentials caused by electrochemical interactions between the electrode and the earth.

Potential - Voltage.

Self-potential method - A passive electrical exploration method in which spontaneous potentials are measured. Syn: spontaneous potential method.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that the project-specific plans are in accordance with these procedures, where applicable, or that other approved procedures are developed. The Project Manager is responsible for ensuring that the personnel operating and interpreting the geophysical data are trained, skilled in that endeavor, so far as to receiving documentation on the training and experience of the operating personnel.

Field Team Leader - The Field Team Leader is responsible for selecting and detailing the geophysical techniques and equipment to be used. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the activities have been briefed and trained to execute these procedures.

## 5.0 PROCEDURE

### 5.1 Overview

Self-potential (SP) surveys are used to detect geochemical activity and groundwater seepage. SP data consist of measurements of naturally occurring potentials across two electrodes placed on the earth's surface. Potentials measured during these surveys are small, generally less than 100 millivolts, and may be positive or negative.

Sources of SP effects are varied and include oxidation of sulfide mineral deposits, bioelectric activity in vegetation, varying electrolytic concentration in water, fluid motion through a cavity (known as streaming potentials), and a variety of other meteorological (e.g., thunder storms) and geochemical sources. SP surveys are most often used for relatively shallow exploration, and are interpreted only qualitatively. The non-uniqueness of SP sources requires that SP surveys should always be augmented by other geological or geophysical data. General discussions of the SP technique may be found in Dobrin (1976) and Telford et al., (1976).

### 5.2 Applications

The two most likely SP survey applications in site assessment studies are:

- Locating groundwater seepage zones
- Locating near surface inorganic contaminants

Leakage from lagoons which hold electrolytic solutions has been successfully identified by the SP method, and examples may be found in Bogoslovsky and Ogilvy (1972, 1973), and Ogilvy and Bogoslovsky (1969, 1979).

### 5.3 Equipment

Equipment needed to perform SP surveys includes:

- A high-impedance millivolt meter.
- Non-polarizing electrodes, minimum of two
- Connecting cables

The millivolt meter can consist of either a digital multimeter (commonly used in electronics diagnosis and repair) or a voltmeter intended solely for SP measurement. Either of these instruments should have a high input impedance to avoid drawing excessive current from the earth, and they should be mounted in a non-grounding case.

Non-polarizing electrodes must be used because a standard metal electrode will develop a self-potential of its own when placed in the earth. Non-polarizing electrodes are commonly of the porous-pot type, consisting of an unglazed ceramic pot containing a metal electrode and a saturated electrolytic solution. The solution must contain a salt of the same metal as the central electrode; e.g., a solution of copper sulfate is often used with a copper electrode. The porous pot is placed on the ground surface, and electrical contact with the earth is achieved by seepage of the electrolytic solution through the porous ceramic. The electrode is connected to the millivolt meter by insulated wire. Wire lengths may extend several thousand feet, depending on the area to be surveyed and the electrode array used.

## 5.4 Field Procedures

SP measurements are performed either along linear traverses or along the nodes of a survey grid. If possible, it is desirable to orient the traverses perpendicular to the trend of the suspected SP source. For example, leakage from a lagoon is most effectively evaluated by SP traverses parallel to the sides of the lagoon. Electrode positions should be determined with a non-conductive measuring tape, usually fiberglass, to avoid providing an unintended current path.

Two electrode arrays may be used. For the first, one electrode is placed at a base station while the other electrode is moved to measurement locations. Potentials are then measured with respect to the same point, the base station, producing data for profiling. The great lengths of wire needed to reach measurement stations far from the reference electrode may necessitate more than one base station for large sites.

The second electrode array involves moving both electrodes while maintaining a constant electrode separation; this procedure also requires a sequented use of measurement positions such as 1-2, 2-3, 3-4 etc. This electrode array minimizes the lengths of wire needed, but can introduce cumulative errors. Multiple traverses completed with this electrode array must be tied together by measuring potentials between the lines.

## 5.5 Interpretation

### 5.5.1 Data Analysis

The SP data is contoured or plotted as profiles, depending on whether data were collected along the nodes of a grid or as individual traverses. Profiles are constructed with distance along the traverse on the x-axis and the SP measurement on the y-axis of standard graph paper. The data plots are examined for variations in SP values that may indicate the target of interest.

### 5.5.2 Presentation of Results

SP results are displayed in the form of either contoured or profiled SP voltage measurements, and referenced to plan maps for position and cultural features.

### 5.5.3 Interpretation of Results

Interpretation of SP data is highly subjective. Areas of SP values which differ from the apparent background values are identified and correlated with other data sets. Depending on the target of interest, the anomaly may be either positive or negative in polarity.

SP is best used in conjunction with other techniques. The type of intended target will determine the other geophysical technique to be used; electromagnetic terrain conductivity, ground penetrating radar, and electrical resistivity measurements are most common.

## 5.6 Advantages and Disadvantages

The primary advantage of SP surveying is low cost, due to the inexpensive equipment used. In addition, SP is one of the few geophysical techniques that can detect leakage positions and paths.

The disadvantages are the non-uniqueness of the method, because of the many possible SP sources, and the highly subjective nature of SP interpretation.

## 6.0 QUALITY ASSURANCE RECORDS

All field data will be recorded in Field Logbooks with the date, location name, personnel conducting the investigation, start time and ending time (all times in military time).

## 7.0 REFERENCES

Bogoslovsky, V.V. and Ogilvy, A.A., 1972, The Study of Streaming Potentials on Fissured Media Models: Geophysical Prospecting, v. 20, no. 4, p. 109-117.

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# F708
# *GRAVIMETRY*

# GEOPHYSICS: GRAVIMETRY

## 1.0 PURPOSE

The purpose of this SOP is to provide general reference information for using gravimetric methods.

## 2.0 SCOPE

This SOP indicates a description of field procedures, equipment, and interpretation methods necessary to fully utilize this procedure.

## 3.0 DEFINITIONS

Bouguer slab - A slab of infinite horizontal extent, constant density and thickness

Bouguer anomaly - Gravity value after the observed (measured) gravity has been corrected for latitude, free-air, Bouguer slab and terrain.

Complete Bouguer anomaly - Gravity value after the observed (measured) gravity has been corrected for latitude, free-air, Bouguer slab and terrain.

Simple Bouguer anomaly - Gravity value after the observed (measured) gravity has been corrected for latitude, free-air, and Bouguer slab.

Earth tides - Variations in the gravitational attraction of the sun and the moon as their positions change with respect to the earth, (maximum amplitude 0.3 gal occurring in a period as short as an hour).

Residual gravity map - resulting gravity map after regional gravity affects are removed from Bouguer anomaly values.

The international gravity value - Equation that accounts for the fact that the earth is not a perfect sphere, but is more like a perfect fluid for which balance is maintained between the gravitational forces tending to make it spherical and the centrifugal forces of rotation tending to flatten it. As a result, the equatorial radius is approximately 21 km greater than the polar radius.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that the project-specific plans are in accordance with these procedures, where applicable, or other approval procedures are developed. The Project Manager is responsible for development of documentation of procedures which deviate from those presented herein.

Field Team Leader - It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the activities have been briefed and trained to execute these procedures.

## 5.0 PROCEDURES

### 5.1 Overview

The gravity survey method is a passive - geophysical technique which measures extremely small variations in the earth's gravitational field, using a highly sensitive instrument. Observed gravity measurements are variations of the earth's true gravitational attraction from one location on the earth's surface to another (Dobrin, 1960). Spatial variations in the value of observed gravity depend upon a number of factors including:

- Lateral density variations of earth materials in the vicinity of an observation point
- Elevation
- Latitude
- Surrounding terrain variations (topography)
- Tidal fluctuations

In gravity exploration the variation in density is the only significant factor (Telford, 1978). Lateral variations in the distribution of mass in the earth's crust produce distortions or differences in the gravitational field. Tectonics, faulting, erosion, deposition and other geologic movement involving rock often result in lateral density variations in the subsurface rocks. Measured gravitational differences are interpreted in terms of probable subsurface mass distributions, which are inferred from surface and near surface geologic conditions (Nettleton, 1975).

The acceleration of gravity at the earth's surface is approximately 980 centimeters per second squared or 980 gals. In gravity exploration work, anomalies as small as one ten - millionth of the earth's field are detected with gravimeters. The unit used in exploration gravity surveying is the milligal ($10^{-3}$ gals). Microgravimeters are more sensitive instruments which can detect smaller magnitude anomalies, measured in the unit of microgals ($10^{-6}$ gals).

### 5.2 Applications and Uses

After the appropriate corrections have been made, gravity values can be presented as Bouguer anomalies. A Bouguer anomaly map (Figure 1) looks very much like a topographic contour map. Bouguer anomalies are interpreted in terms of the size, shape and position of the subsurface structures. Microgravity measurements can be used to detect the following conditions:

- Joints
- Dissolutions
- Collapses
- Cavities
- Buried river channels
- Fault scarps

Field Team Leader - It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that the field investigation personnel performing the activities have been briefed and trained to execute these procedures.

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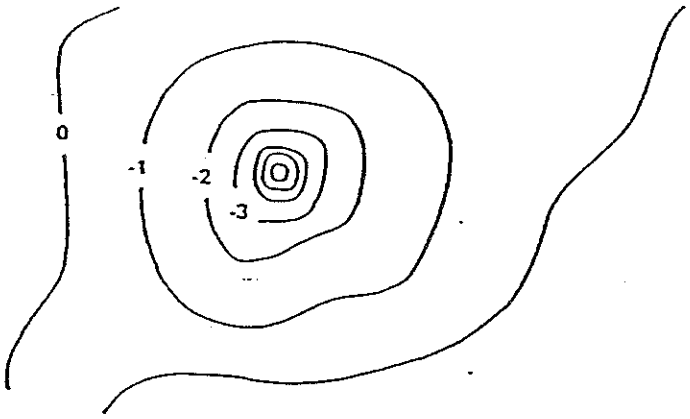
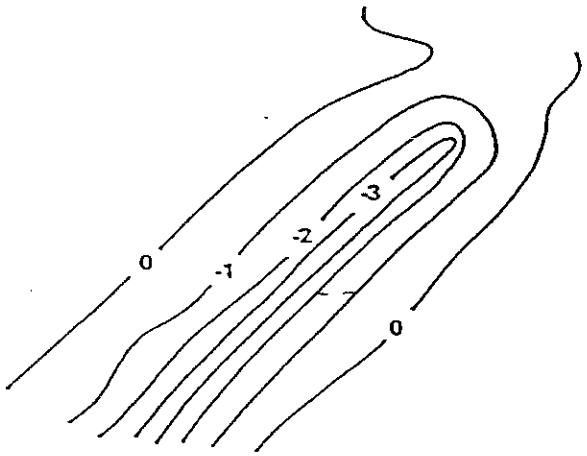
FIGURE 1






A.) GRAVITY ANOMALY OF SPHERICAL CAVITY




B.) GRAVITY AND ANOMALY OF HORIZONTAL CYLINDRICAL CAVITY

Source: Butler, 1977

CONTOUR MAPS ILLUSTRATING NEGATIVE GRAVITY ANOMALIES

## 5.3 Equipment

The Lacoste - Romberg and Worden gravimeters are two commercially available gravimeters. The Lacoste - Romberg Model D is the only commercially available microgravimeter. These instruments measure the elongation of a spring which supports a weighted beam. An increment of elongation of the spring is proportional to an increment of gravity. The principal of operation of a Lacoste -Romberg microgravimeter is illustrated in Figure 2.

The Lacoste-Romberg gravimeter is heated to maintain a constant instrument temperature and, consequently, a more sensitive and stable reading. Some instruments are not temperature controlled and, consequently, instrument temperatures must be noted and corrected for. Gravimeters with heaters require a portable energy source (batteries) and must have an appropriate warm up time (approximately one day) to acquire stable, accurate readings.

## 5.4 Field Procedures

The gravimeter itself can be operated by a single operator. The topographic survey can be performed before, during, or after the gravity measurements.

The operation of the instrument is rather straightforward, but acceptable levels of accuracy require meticulous attention to details, such as:

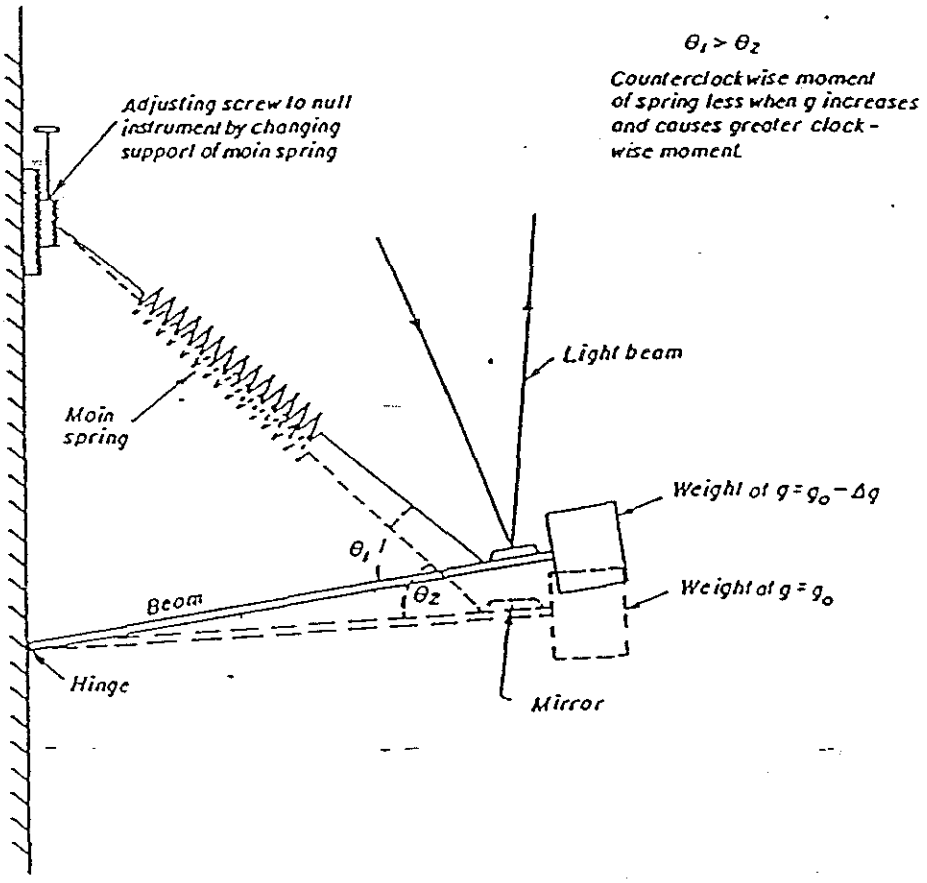
- Instrument leveling
- Surveyed location
- Surveyed elevation
- Instrument drift
- Time of measurement

Survey accuracies of at least one-tenth of a foot of elevation, and horizontal accuracy of approximately 2 feet are required for microgravity surveys. The high level of accuracy required in gravity surveys dictates repeated readings at a base station throughout the period of the survey to compensate for time variations (drift) inherent in all instruments. Typically, base station readings are taken at least three times a day and often are repeated in one-hour intervals. Loop times for microgravity instruments that have been recently reheated to operating temperatures initially should be at about 30 minutes intervals. Loop times can then progress up to a maximum of 1 hour. Initial short loop times are to minimize errors due to mechanical adjustments caused by internal thermal stress. Microgravity instruments with fluid levels should have 30 second levels rather than 60 second levels. The 30 second levels provide a more precise leveling accuracy.

At each station the gravimeter is set on a metal tripod which provides a stable base. The instrument is leveled by two horizontal and mutually-perpendicular levels in the instrument. Three readings are commonly taken at each station. Levels checked between readings to ensure data quality and minimize operator error.

FIGURE 2






OPERATION OF LACOSTE AND ROMBERG GRAVIMETER (SCHEMATI

Field Logbooks

Important information that should be recorded in ~~field note books~~ are:

- Instrument identification number
- Date and time of reading
- Operator
- Station identification number
- Base station location
- Instrument readings

To assure correlation between data sets, the relative gravity for each base station can be established by looping with absolute base stations which are part of an international gravity network adjusted to the 1979 Potsdam value. A listing of absolute base station locations can be obtained from NOAA in Washington, D.C.

Gravity stations are arranged either in gridded survey patterns or in linear traverses. Gridded survey patterns provide more detailed information, but at a higher cost due to the higher number of stations.

Precautions should be taken in locating gravity stations to avoid whenever possible areas with nearby (50+ feet) severe topographic changes. These topographic changes affect gravity measurements. Hills rising above the gravity station and valleys (lack of material) affect the gravity measurements by reducing the recordings due to upward attraction of hills or lack of downward attractions by a valley (Telford). Earthquakes and other vibratory phenomena also can adversely affect gravity readings. Generally, when an ur ake has occurred the instrument reading beam may drift and cannot be stabilized. Depending on the sta..e to and magnitude of the earthquake, gravity measurements should be suspended for a few hours or until the next day. Another potential problem in conducting a gravity investigation is unstable ground aterials such as loose sand, nearby sources of vibration (heavy truck traffic, construction equipment, etc.) stone ballast. Generally, these situations can be overcome with patience, slight changes in station locations altering the time of day in which the investigation is conducted.

## 5 Interpretation

### 5.1 Data Analysis

Gravity data obtained in the field must be corrected for:

- Instrument drift
- Earth-tide variations
- Elevation
- Bouguer slab
- Latitude
- Influence of surrounding topographic (terrain) variations

All gravity stations within a common data set must be reduced to a common elevation datum plane. Sea level is the most often used datum plane.

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- Instrument identification number
- Date and time of reading
- Operator
- Station identification number
- Base station location
- Instrument readings

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All gravity stations within a common data set must be reduced to a common elevation datum plane. Sea level is the most often used datum plane.

5.5.1.1 Instrument Drift and Earth-Tide Variations

The observed gravity for each station is determined by looping with a base station with a known gravity value and correcting readings for instrument drift and earth tide variations. Instrument drift and earth tide variations are calculated by dividing the difference in the base station readings (end of loop minus beginning of loop) by the time required to complete the loop. Each station reading is then corrected by adding the drift factor by the time between stations. Observed gravity values are then calculated by multiplying the corrected meter reading difference between the base and the gravity station by factors unique to the particular gravity meter.

5.5.1.2 Elevation Correction

Since gravity varies inversely with the square of distance, it is necessary to correct for changes in elevation. Correction for elevation or the free-air correction compensates for elevation variations between stations so that all the field readings are reduced to common datum surface. Sea level is the common datum surface used for the free-air corrections. When the gravity stations are above the datum plane, the free-air corrections are added to the observed gravity values (Telford, 1978).

5.5.1.3 Latitude Corrections

Latitude corrections compensate for the centrifugal acceleration due to the rotation of the earth and radius variation between the poles and equator. Maximum latitude corrections occur at latitude 450 where the variation is approximately 0.01 milligals per 40 feet of north--south displacement.

The International Gravity Formula of 1967 incorporates the latitude correction in the calculation of theoretical gravity. When gravity stations are north of the reference latitude, all corrections are subtracted from the observed gravity values (Telford, 1978).

5.5.1.4 Bouguer Slab-Correction

The free-air correction accounts for elevation differences, but not for attraction of the material between the station and the common datum surface. The Bouguer correction accounts for the attraction of this material. Bouguer corrections assume a slab of infinite horizontal extent.

A commonly used slab density in New England is 2.67 g/cm , which is the approximate density of the granitic crust. Bouguer corrections are applied in the opposite sense of the free-air corrections; that is, they are subtracted when the station is above the common datum plane (Telford, 1978).

5.5.1.5 Terrain Correction

Terrain corrections are applied to the gravity data when the topography of the surveyed area is not relatively flat. Nearby hills result in an upward component of gravity and nearby valleys result in an apparent loss of mass between the station and datum elevation. Both effects diminish the measured gravitational field, therefore, the terrain correction is always added to the data. Terrain corrections are calculated using the slab density used in the Bouguer slab correction. There are several graphical methods for calculating terrain corrections. All require a good topographic map of the area at a minimal 10-foot contour interval. The most commonly used graphical method uses templates that divide the area into zones for which the average

elevation can be estimated and the terrain correction calculated. Tables of terrain corrections, for zone charts of particular dimensions developed by Hammer (1939) facilitate this operation considerably (Telford 1976).

5.5.1.6 Theoretical Gravity

The difference between the corrected station gravity and the calculated theoretical gravity for each station is the Bouguer gravity. Theoretical gravity values are calculated using station latitudes and a relationship adopted by the International Association of Goedesy.

### 5.5.2 Presentation of Results

The results of a gravity survey can be presented as contour maps or as profiles depending upon the data processing and/or interpretation techniques. A raw-data map presents the gravity readings that have been corrected for instrument drift and earth-tide effects. A free-air gravity map is the raw data corrected for station elevations (reduced to a common elevation datum). A simple Bouguer map is the free-air gravity values corrected for the Bouguer slab. A complete Bouguer map is the simple Bouguer values corrected for terrain variations. Residual anomaly map is the residual gravity values after regional gravity affects have been separated. Data processing procedures to prepare each of the above mentioned maps include assumptions that may or may not be true and may bias the interpretation of the gravity data. Therefore, the preparation and qualitative analysis of these maps may be necessary to identify any bias or anomalies that may be created due to the data processing calculations.

Interpreted gravity results are presented as 2-D, 2 1/2-D or 3-D profiles or maps. The 2-D results assume infinite lengths in the 3rd dimension, 2 1/2-D results have a finite length in the 3rd dimension and 3-D modeling results have 3 dimensional geometric shapes.

### 5.5.3 Interpretation

The complete Bouguer anomaly map represents the contribution of all earth materials that exist beneath the ground's surface. Therefore, the first step in the interpretation of gravity data is to separate the anomaly components arising from the source of interest from the sources of no interest. This step is called the regional residual separation and it is the residual values that are of primary interest.

The residual or shorter wavelength gravity data can be separated and modeled independent of the deeper-seated, longer wavelength regional gravity data. The residual data can be separated from the regional data in a number of ways. The averaging method, polynomial fitting, and upward continuation and wavelength filtering regional residual separation methods are a few. Text books such as Telford (1976), Nettleton (1976) etc. explain in detail regional residual separation methods.

There is extensive literature on the subject of and significant problems involved in regional residual separation. The techniques listed above are some of the more common techniques used. The choice of the method used for removing the regional, depends upon many factors, the most important being the total labor involved, the complexity of the gravity map, the density and distribution of the stations and quality of the data.

The residual gravity maps are a by-product of the regional residual separation. These maps are used to predict the physical characteristics and proximity of near surface anomalous bodies.

Before a quantitative interpretation is attempted, a qualitative analysis of the data should be made to determine the presence of anomalous sources and to get a general idea of depth, strike, and density of sources. Qualitative analysis includes an evaluation of the polarity, magnitude, gradient and trends of residual anomalies, as well as a comparison with other available geophysical (magnetic, seismic, electrical) and geological data.

Based on the qualitative analysis of the regional map a quantitative interpretation to determine possible individual sources of the anomalies can be undertaken. The quantitative interpretations are accomplished using 2 and 3 dimensional computer modeling techniques. Each anomaly body is assigned a geometric shape and a value which represents the bodies contrasting density values. All gravity interpretations benefit from incorporation of geologic constraints. Such constraints can come from surface geology, geomorphology, subsurface geology, boring logs, seismic reflection and refraction data, magnetic surveys, and geochemical data.

Interpretation of gravity data is subject to two limitations:

- The inherent ambiguity in the possible source of a given gravity anomaly.
- The complete dependence of gravity anomalies on the existence and magnitude of horizontal variations in the density of the rocks.

For a given distribution of gravity there is no single, unique distribution of mass which will have a calculated effect that corresponds to the observed gravity. That is, for a given width of anomaly there is a corresponding maximum depth and a cone of possible sources, as illustrated in Figure 3.

## 5.6 Advantages and Limitations

The advantages of a gravity survey are:

- Field work can be carried out by one to three persons in any accessible area, including highly developed urban and industrialized sites, over pavements, fills, landfills, on lake ice, inside buildings, etc.
- Instrumentation is portable, the work can be silent and produce no visible disturbance to an environment other than stakes or other station markings.
- The method lends itself well to areal coverage. Contour maps of bedrock or other features have obvious advantages over information at points or along profiles.
- Used appropriately, it is highly cost effective either by itself or in combination with other exploration methods.

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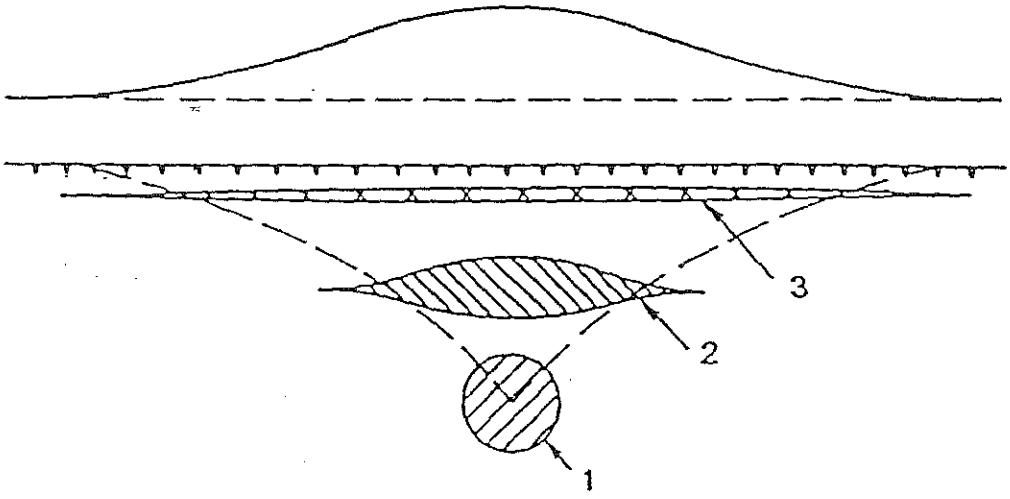
FIGURE 3






Gravity anomaly shown can be approximated by a deep sphere (1) or by a shallower broader body, such as (2) or (3) [from Nettleton, 1976]

CONE OF INFLUENC

The limitations of a gravity survey are:

- Applications are limited to mapping of density - dependent interfaces.
- Accurate station locations and elevations are necessary.
- Calibration with geological "knowns" such as outcrops, borings, seismic profiles, etc. is necessary for quantitative work.
- Excessive topography, access problems, and certain bedrock complexities may seriously limit the accuracy of data interpretation.

## 6.0 QUALITY ASSURANCE RECORDS

All data will be recorded in Field Logbooks under the following format: date, start and end time (military time), personnel on site and weather.

## 7.0 REFERENCES

Beck, A.E., 1981, Physical Principles of Exploration Methods: London, MacMillan Press LTD.

Dobrin, M.B., 1976, "Introduction to Geophysical Prospecting," New York, McGraw-Hill, 630 p.

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Nettleton, L.L., 1973, Elementary Gravity and Magnetics for Geologists and Seismologists: Tulsa, Oklahoma, Society of Exploration Geophysicists, Monograph no. 1, 121 p.

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Talwani, M., Worzel, J.L., Landisman, M. 1959, Rapid Gravity Computations for Two-Dimensional Bodies with Application to the Mendocino Submarine Fracture Zone: Journal of Geophysical Research, v. 64, no. I, p. 49-57.

Telford, W.M., Geldart, L.P., Sheriff, R.E., and Keys, D.A., 1976, Applied Geophysics, New York, Cambridge University Press, 860 p.

Texas Instruments, 1982, Worden Gravity Meter Operating Instructions: Houston, Texas.

Van Balauicom, Richard, 1980, Practical Geophysics: Northwest Mining Association, 1980.

# F709
# *SITE UTILITY CLEARANCE PROCEDURES*

## 1.0 PURPOSE

The following SOP provides a description of site utility clearance procedures Baker representatives will implement prior to beginning field activities. This utility clearance is designed to minimize the risk to personnel from striking a potentially dangerous (usually hidden) obstacle.

## 2.0 SCOPE

This SOP provides the procedures to be taken by Baker personnel in locating on-site utilities. Baker personnel may become aware of site specific conditions that may require deviation from the normal SOP.

## 3.0 DEFINITIONS

It is the responsibility of the Baker representative to coordinate arrangements with the facility contact to have a site utility clearance conducted at the site prior to the first day of field activities. Additional responsibilities include being present during this clearance and later briefing all on-site Baker personnel of the results of this meeting.

## 4.0 RESPONSIBILITIES

Baker Representative - The Baker representative will include, but not be limited to, Project Managers, Project Geologists, Environmental Scientists, and Technicians.

Utilities - This term will include, but not be limited to, the following:

| | | |
|---|---|---|
| Electric | Fuels | Sewers |
| Water | Steam | Storm Drains |
| Telephone | Gas | |

## 5.0 PROCEDURES

This section reviews the procedures and tasks required of the Baker representative to conduct a site utility clearance.

- Arrange a meeting with the facility contact and facility utility personnel prior to the first day of scheduled field activities.
- Review files obtained from the site visit and/or other previous investigations for information regarding the site.

### 5.1 After Site Arrival

The following tasks will be completed upon arrival at the facility.

a. The Baker representative and facility contact will proceed to the site and complete Section I of the Site Utility Clearance Checklist. (See attached copy.)

b. The Baker representative, along with facility utility personnel, will begin locating underground utilities using visual inspection, drawings and maps, equipment, and personal knowledge.

c. Smaller work areas will have all utilities clearly marked on the ground with fluorescent paint indicating direction and type of utility. Surrounding areas also will be cleared to allow for flexibility in placement of boring locations.

d. Larger sites will have a boring location marked with fluorescent paint. Utility personnel will be instructed to clear the spot as well as mark any utilities that fall within a 10 ft. radius. Directional trends of utilities will be reviewed to provide routes of such utilities.

e. Upon completion of the meeting, the Baker representative will check the cleared locations with their own equipment. This equipment is meant to provide a verification; it is not to be used as a primary means of utility detection.

f. Locations of utilities (both overhead and below ground) will be noted and included on a map or a sketch in the Field Notebook.

## 6.0 QUALITY ASSURANCE RECORDS

Quality Assurance Records shall consist of a complete site utility clearance checklist and accompanying site sketch.

## 7.0 REFERENCES

None.

# SITE UTILITY CHECKLIST

**Site Name** _____ **Date** _____

**Project Number** _____ **Baker Rep** _____

| UTILITY | CLEARANCE GROUP REP. | CLEARANCE METHOD | BAKER CLEARANCE CHECK | COMMENTS |
|---|---|---|---|---|
| Telephone | | | | |
| Electric | | | | |
| Fuel | | | | |
| Natural Gas | | | | |
| Steam | | | | |
| Water | | | | |
| Sewer | | | | |
| Storm Drain | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Was the facility representative present during the utility clearance? Yes _____ No _____
Was the driller present during the utility clearance? Yes _____ No _____
Are there overhead utilities present? Yes _____ No _____
Additional Comments:

# F801
# *LAND SURVEYING*

# LAND SURVEYING

## 1.0 PURPOSE

This procedure describes methods and equipment commonly used by a Registered Land Surveyor when compiling by survey the vertical and horizontal locations of on-site monitoring wells and other site structures, and topographic features associated with study areas at various locations.

## 2.0 SCOPE

The information presented in this SOP is generally applicable to various locations, except where state-specific requirements differ concerning certifications, licenses and registrations.

Specific surveying problems encountered by the survey crew may require the adaptation of existing equipment or design of new equipment. Such innovations shall be documented in the survey crew's Field Logbook.

## 3.0 DEFINITIONS

North American Datum (AND) - Datum used during the absence of established horizontal and vertical control.

Mean Sea Level (MSL) - Adopted as a datum plane for the measurement of elevations and depths.

Horizontal Control - Horizontal location of an object from surveyed corners or other features on permanent land monuments in the immediate site area. Will be based on North American Datum (AND).

Vertical Control - Vertical location of an object compared to the adjacent ground surface.

Bench Mark - Precisely determined elevation above or below sea level. May also have horizontal control (northing, easting) determined for location.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with acceptable surveying practices as required by the state in which the work is performed.

Field Team Leader - The Field Team Leader is responsible for ensuring that procedures are implemented in the field and that personnel performing surveying activities have been briefed and trained to execute these procedures.

## 5.0 PROCEDURES

The services of a Registered Land Surveyor will be required to determine by survey the elevations and horizontal locations of monitoring wells and other site structures, and topographic features associated with study areas at various locations. The surveyor will mobilize to the site within seven days upon receiving the notice-to-proceed. All site surveys must be completed in the time frame agreed upon. The site map shall be completed within ten days of completion of the site survey.

5.1

Specifically, Baker requires the following will be required:

Delineate the elevations of groundwater monitoring wells to an accuracy of 0.01 feet, referenced to United States Geological Survey Mean Sea Level (MSL) from the nearest datum bench mark.

5.1.1

The elevation point for each well casing and a permanent mark designating the elevation point shall be established on each well. In addition, the ground surface elevation for each well shall be established. Some of the wells will be flush-mounted level with the pavement; thus the land surface elevation will be above the "top of casing" elevation for those wells.

5.1.2

Determine the elevation of the directly adjacent ground surface to an accuracy of 0.1 feet.

5.2

Delineate the horizontal location of each well from surveyed comers or other features on permanent land monuments in the immediate site area to an accuracy of 0.1 foot, referenced to North American Datum (AND). Baker will supply an existing property plot plan or CADD file for each site to the subcontractor that will serve as the base map for locating surveyed points. All permanent points established during control traverses shall be shown.

5.3

Locate various drainage trenches/structures and significant topographic features at Baker's request via the survey. An Alliance and/or a U.S. Steel representative may be present during survey activities to identify points and features to be located. If no Baker representative will be present, the subcontractor will be notified in advance as to what features or types of features are to be included in the survey.

5.4

In the absence of facility-established horizontal and vertical control, all survey points will be based on North American Datum (AND) for horizontal control and MSL for vertical control.

5.5

The subcontractor shall provide a letter report containing all relevant survey information along with one legible copy of the field survey notes recorded when determining the surveyed elevations, location of wells, and requested topographic information. The subcontractor shall also provide one reproducible, legible copy of the property map showing the well designation, "top-of-casing" elevation and location at each well, and a table listing the well designation, "top-of-casing" ground surface elevations, coordinates for each well, and plotted horizontal features. The accuracy of other site maps cannot be verified. It is recognized that the subcontractor's responsibility in plotting features is to provide most accurate locations possible on mapping available. Tabulated data provided by the subcontractor, however, must be accurate on an MSL datum specified above. All deliverables must be in ACAD R12.

5.6

The subcontractor shall perform these services in accordance with standard, acceptable surveying practices as required by the state in which the work is performed and all work shall be conducted under the supervision of a Registered Land Surveyor, duly licensed to work in the state.

## 6.0 HEALTH AND SAFETY

The subcontractor is to provide for and assume responsibility for adequate health and safety protection for on-site personnel. Contracted land surveyors are required to provide evidence of having received OSHA-specified training to conduct work on potentially hazardous sites. The specific content of the training requirements are outlined in 29 CFR 1910.120(e). These requirements include:

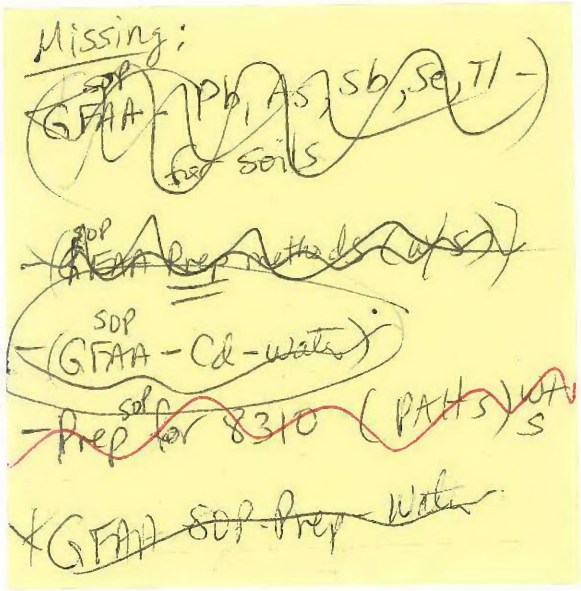
- Minimum of 24 hours of hazardous waste training
- Eight hours of additional training for supervisors
- Eight hours of hazardous waste refresher training for every year after the initial 24 hour training
- Medical surveillance as specified in the specific OSHA regulations

At least one of the on-site surveying personnel must have the 32 hour supervisor hazardous waste training. The subcontractor is to provide copies of current training and medical certifications, and to assure that this documentation accompanies their personnel onto the job site.

## 7.0 QUALITY ASSURANCE RECORDS

The Field Logbook shall serve as the quality assurance record for on-site surveying activities.

APPENDIX D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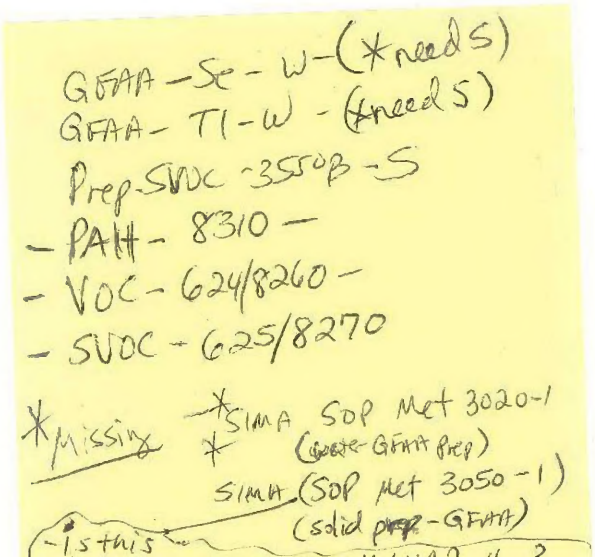
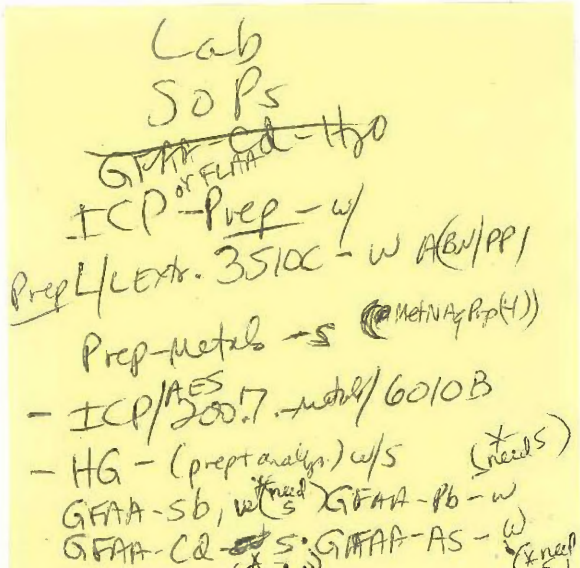
SOP
GFAA - Cd - Water
Prep SOP for 8310 (PAHs) Waters
*GFAA SOP Prep Water


GFAA - Se - W - (*needs)
GFAA - Tl - W - (*needs)
Prep-SVOC - 3550B - S
- PAH - 8310 -
- VOC - 624/8260 -
- SVOC - 625/8270
*Missing
* SIMA SOP Met 3020-1 (waste-GFAA Prep)
SIMA (SOP Met 3050-1) (solid prep-GFAA)
- is this


Lab
SOPs
GFAA - Cd - H2O
or FLAA
ICP - Prep - W
Prep L/L Extr. 3510C - W ABN/PP
Prep-metals - S (Method Prep(H))
- ICP/AES 200.7 - metals/6010B
- HG - (prep + analysis) w/S (*needs)
GFAA - Sb, W (*needs) GFAA - Pb - W
GFAA - Cd - S; GFAA - As - W (*needs)

# STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL OR DISSOLVED METALS ANALYSIS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY

Originating Author: Karin Stewart
Revision Author: Troy Goehl

This SOP is effective upon signed approval by the following:

Troy Goehl
Unit Supervisor

12-13-2001
Date

Jeffrey M. Farrow
QA/QC Director

12-13-2001
Date

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

# 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This procedure is based EPA Method 200.0 section 4.1.3 and SW-846 Method 3020A. This procedure is applicable to the digestion of all aqueous samples and the extracts from the TCLP and SPLP procedures. This procedure is not applicable for the preparation of samples to be analyzed by Inductively Coupled Plasma Emission Spectroscopy or Flame Atomic Absorption Spectroscopy.

## 3.0 SUMMARY

3.1 1 ml of 30% hydrogen peroxide and 1 ml of concentrated nitric acid are added to a 50 ml aliquot of sample. The mixture is heated near boiling for 1 hour. An additional 1.5 ml of nitric acid is added to the sample and the mixture is again heated to near boiling. The sample is then cooled and diluted to a final volume of 50 ml with DI water.

3.2 This procedure is a combination of the EPA methods referenced in section 17.0. There are differences in the acid volumes and digestion times used in the reference methods. This procedure uses a 50 ml aliquot of sample, whereas the reference methods are based on 100 ml. Moreover, this procedure involves no concentration or dilution based on the initial and final sample volumes, whereas the reference methods include a 2:1 concentration of the initial sample size. The volumes of acid used have been adjusted accordingly and, where different volumes of acid are listed in the reference methods, the average of the two is used. The reference methods include the use of hydrogen peroxide for the preparation of samples analyzed for Arsenic or Selenium only. This procedure includes the peroxide regardless of the elements measured. These changes from the written methods are considered acceptable as shown through the continued generation of acceptable control samples and performance evaluation samples.

## 4.0 DEFINITIONS

4.1 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.2 Analyte – The specific component measured in a chemical analysis.

4.3 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Calibration Blank – An aliquot of the standard diluent (water or organic solvent) that is not carried through the sample preparation scheme. It is analyzed to verify that the analytical system is free from contamination. Also referred to as an instrument blank or solvent blank.

- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.

- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Preparation or Procedural Blank.

4.4 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.5 Laboratory Control Sample (LCS) – An aliquot of clean matrix (lab pure water or vendor supplied solid) spiked with target analytes or compounds representative of target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.6 Laboratory Control Sample Duplicate (LCSD) – An aliquot of laboratory pure reagent spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.7 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.8 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.9 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.10 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.11 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.12 Sample – A portion of material supplied by the client for analysis.

4.13 Sample Duplicate – Two aliquots of the same sample processed independently. This monitors precision of the analysis. Precision results are reported as relative percent difference (RPD).

## 5.0 INTERFERENCES

5.1 The digestion procedure may not be sufficient to completely break down some metal complexes. Additional digestion time or a more vigorous digestion may be required to facilitate complete digestion. Typically, complete oxidation is evidenced by a light color or no change upon addition of acid or continued heating.

5.2 Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Labware suspected of causing contamination should be soaked with 1:5 nitric acid and rinsed thoroughly with lab pure water.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Digestion vessels, Environmental Express catalog #SC475 or equivalent

7.3 Filters, Environmental Express FilterMate, catalog #SC0401, or equivalent

7.4 Ribbed watch glasses, Environmental Express catalog #SC505 or equivalent

7.5 100 ml graduated cylinders

7.6 Digestion block capable of maintaining 90 to 95°C

7.7 Class A volumetric pipets

7.8 Oxford-style repipetters

7.9 Vacuum filtration device

7.10 0.45 micron filters

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

8.2.1 Lab pure water. ASTM Type II water is generated in accordance with the procedure described in the Quality Assurance Plan.

8.2.2 Nitric acid, conc. $HNO_3$: Trace metals grade (Fisher, AS09-212 or equivalent)

8.2.3 Hydrogen Peroxide, 30% $H_2O_2$: Fisher, H325-500 or equivalent

8.3 Standards

8.3.1 Stock Spiking Standard: Inorganic Ventures #CLPF-ASPK-1 or equivalent contains the following constituents. Store this standard in the standards cabinet located in the metals instrument lab.

| VENDOR | CATALOG # | ELEMENTS | CONC., ug/ml |
|---|---|---|---|
| Inorganic Ventures | CLPF-ASPK-1 | Sb | 120 |
| | | As, Pb, Tl | 20 |
| | | Cd, Se | 10 |

8.3.2 Chromium Stock Standard, 1000 ug/ml. Store this standard in the standards cabinet located in the metals instrument lab.

8.3.3 GFAA Spike Solution: In a 100 ml volumetric flask, dilute 10.0 ml of the stock spiking standard and 0.20 ml of the stock chromium standard to the mark with 5 ml conc. $HNO_3$, and DI water. This prepares a standard of 12 ug/ml Sb, 2 ug/ml As, Cr, Pb, Tl, and 1 ug/ml Cd and Se. Store this standard in the metals sample preparation area.

8.3.4 LCS/MS: Add 1 ml of the GFAA spike solution to 50 ml DI water or sample to prepare the LCS or MS, respectively. This prepares a LCS/MS of 240 ug/l Sb, 40.0 ug/l As, Cr, Pb, Tl, and 20.0 ug/l Cd and Se.

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Samples should be collected in a plastic container. Preservation consists of $HNO_3$ to pH < 2. For Dissolved Metals, the sample must be filtered through 0.45 micron filter paper prior to acidification.

9.3 Digestion must be performed within the maximum allowable hold time of 180 days from collection.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed by each analyst prior to unsupervised sample preparation and whenever substantial change has occurred in the procedure. Prepare four separate Laboratory Control Standards. These standards must be from a source different from that used for instrument calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.2 A *Method Blank* must be prepared with each batch of maximum 20 samples and at a minimum of one per day. Also, the TCLP/SPLP method blanks must be prepared using the same extraction fluid used in the given samples.

10.3 A *Laboratory Control Sample* must be prepared with each batch of maximum 20 samples and at a minimum of one per day. Also, the TCLP/SPLP LCS must be prepared using the same extraction fluid used in the given samples.

10.4 A *Matrix Spike and Matrix Spike Duplicate* must be prepared with each batch of maximum 20 samples and at a minimum of one per day. If insufficient sample is available for the preparation of the MS/MSD, a duplicate LCS (LCSD) or a MS on two separate samples must be prepared.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Repipetters must be verified on a weekly basis. Details of this requirement and the procedure used to verify their operation are in the Calibration of Manual Repipetters SOP.

11.2 Verify the operation of the digestion block. Temperature must be maintained in the range of 90 – 95°C.

## 12.0 PROCEDURE

12.1 Prior to use, rinse digestion vessels, graduated cylinders, funnels, filtration device, and watch glasses with 1:1 nitric acid and 3 rinses of DI water.

12.2 Shake sample vigorously and transfer 50 ml of sample directly into a digestion vessel. For dissolved metals filter an adequate amount using the vacuum filtration device unless sample was filtered in the field.

12.3 Using an Oxford Macro pipet under a fume hood, add 1 ml of 30% $H_2O_2$ and 1 ml of concentrated $HNO_3$ to the sample and swirl to mix.

12.4 Place the digestion vessel in the digestion block for one hour. Do not allow the digestate to boil. Do not allow the vessel to go to dryness. If the vessel goes to dryness, the digestion must be started again with a new aliquot of sample.

12.5 Cool the digestate to room temperature.

12.6 Add 1.5 ml of concentrated $HNO_3$ to the sample and swirl to mix.

12.7 Place the digestion vessel in the digestion block for one hour. Do not allow the digestate to boil. Do not allow the vessel to go to dryness. If the vessel goes to dryness, the digestion must be started again with a new aliquot of sample. If oxidation is not complete after this second addition of acid and the heating process, add additional $HNO_3$ in 1 ml increments until complete. (Complete oxidation can be visually evaluated. The production of a light colored digestate or no change in appearance with continued digestion is typically indicative of complete oxidation.)

12.8 Cool the digestate to room temperature.

12.9 Dilute, in the digestion vessel, the sample volume to 50 ml with DI water and filter if particulate matter is present.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 Sample preparation is documented on the Metals Digestion Log Sheet.

13.2 Enter the preparation data in the LIMS. Details on the procedure for entering analytical data are in the Preparation Batch Data Entry SOP.

## 14.0 METHOD PERFORMANCE

Not applicable.

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Refer to the SIMALABS International Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

## 17.0 REFERENCES

17.1 EPA Methods 200.0, 206.2, 270.2

17.2 SW-846 Methods 3020A, 7060A, 7740

17.3 SIMALABS International Quality Assurance Plan, current revision

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

None

Client: IDEM-BAA 95-30
SOP ID: SOP-MET-7131-1
Rev. Number: 2.0
Rev. Date: March 1, 1996

# Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Cadmium For Aqueous Samples

Author: Karin Stewart
Prepared For American Analytical, Inc.
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7131

Revision # 2.0
Issued: March 1, 1996

| | |
|---|---|
| Immediate Supervisor | Date |
| Second Supervisor | Date |
| QA/QC Officer | Date |
| Analyst | Date |

Effective: March 1, 1996

**CAUTION**

**Disclaimer: This Standard Operating Procedure has been prepared for the sole use of American Analytical, Inc. and may not be specifically applicable to the activities of other organizations.**

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Client: IDEM-BAA 95-30
SOP ID: SOP-MET-7131-1
Rev. Number: 2.0
Rev. Date: March 1, 1996

# GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF CADMIUM FOR AQUEOUS SAMPLES

## LOCATION:

Metals, Metals Laboratory

## REFERENCE:

SW-846, 3rd Edition, Method 7131

## MATRIX:

Water, Leachates

## QUANTITATION LIMIT:

EQL = 2.0 ug/L; MDL = 0.017 ug/L.

## RANGE:

2.0 ug/L to 20 ug/L without dilution

## PRINCIPLE, SCOPE, AND APPLICATION:

Cadmium in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the

vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

## INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

(1) Successively dilute and reanalyze the samples to eliminate interferences.

(2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-bank absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilzation step. If another acid in addition to $HNO_3$ is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source

of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

## SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during digestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestions are to occur under a fume hood.

## SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous and leachate samples are to be collected in 500 ml plastic containers with teflon lined lids, preserved to pH $\leq$ 2 with nitric acid, and cooled to 4°C until digestion. Samples must be analyzed within 6 months of collection.

## APPARATUS:

1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.

2) Zeeman Graphite Tube Atomizer provides power to furnace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 °C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/min.

3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 ul.

4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and provides data manipulation and reporting of sample calculations.

5) Citizen dot matrix printer, prints calibration and sample results.

6) Class A volumetric pipets

7) Class A volumetric flasks

8) Pipets: Microliter, with disposable tips. sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible sources of contamination prior to their use.

9) Analytical balance

10) Disposable glass serological pipets

## ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Furnace optics are cleaned twice weekly. Plumbing connections, and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

## REAGENTS AND CALIBRATION STANDARDS:

1) Deionized water - Type II

2) Nitric Acid - concentrated, trace metals grade (Fisher, AS09-212)

3) Furnace stock calibration standard: Using a Class A volumetric pipet, dilute 1.0 ml Cadmium Stock (Spex, QC-19 - 1000 ppm), and 4.0 ml concentrated nitric acid, to 200 ml with DI water in a volumetric flask. Bring to volume. This will result in a final concentration of 10 ppm Cadmium. Dilute stock calibration standard 1:99 with DI water for daily calibration.

4) Furnace ICV/CCV Solutions: Using a 100 uL micropipet and a 10 mL glass serological disposable pipet, transfer 0.10 ml QC-19 Stock (SPEX, QC-19, 100 ppm) and 2.0 ml concentrated nitric acid to a 100 ml Class A volumetric flask partially filled with DI water. Bring to volume. This will result in a 100 ppb final concentration. Dilute 1:1 for a working concentration of 50 ppb.

5) Cadmium Modifier: Using an analytical balance, weigh 1.00 g $NH_4H_2PO_4$ (Fisher, A684-500) and transfer to a 100 mL Class A volumetric flask. Dilute to volume.

## CALIBRATION PROCEDURES:

A curve consisting of 4 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of $\geq$ 0.995 to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the PQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCV and CCB must meet the above stated criteria for the ICV and ICB.

## SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

## ANALYSIS PROCEDURE:

1.) Turn on monitor, computer, Spectra AA-400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F-6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.

2.) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swap and isopropyl rubbing alcohol. Clean a graphite tube and it's platform using a Kim wipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite tube to the center of the furnace chimney. Swing the toggle lever counter-clockwise in order to close the right-hand housing onto the tube now positioned inside the furnace housing.

3.) Remove rinse bottle and fill to the line with DI $H_2O$. Clean the Blank, Modifier and Standard cups with DI $H_2O$ and 1:1 Nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.

4.) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampling arm will move from it's rinse position to the sample 1 position and than to the sample introduction hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. Use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5.) Open syringe compartment door. Put the syringe clear of it's mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Re-insert the syringe assembly into it's housing and close the compartment door.

6.) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean). The furnace will heat and clean the graphite tube. Press shift and F11 (start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is properly injected onto the platform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it in the path of the UV light and thus putting in view the position of the capillary while inside the graphic tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the analytical signal.

7.) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knobs on the left-hand side of the appropriate lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its

furthest most position from the left-hand baseline.) Note: pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8.) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour check standards into sample cups and place them in their proper positions in the autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for Cadmium:

Cadmium: Program #6, Matrix Modifier - Cadmium Modifier
Standard 1 =0.25 ppb, 2 = 0.50 ppb, 3 = 1.00 ppb, 4 = 2.50 ppb
ICV = 1.00 ppb, CCV = 1.00 ppb

QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within ± 20% of true value.

At least one preparatory blank, laboratory standard, spike and duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See belo

*Method of standard additions:*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

1) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

3) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume $V_x$, are taken. To the first (labeled A) is added a small volume $V_s$ of a standard analyte solution of concentrate $c_s$. To the second (labeled B) is added the same volume $V_s$ of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration $c_x$ is calculated:

$$c_x = \frac{S_B V_S c_S}{(S_A - S_B) V_X}$$

where,

$S_A$ and $S_B$ are the analytical signals (corrected for the blank) of solutions A and B, respectively. $V_S$ and $c_s$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_S$ is made much less than $V_X$, and thus $c_s$ is much greater than $c_x$, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

## DATA TREATMENT:

For determination of metal concentration by direct aspiration and furnace; read the metal valu ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

$$\text{ug/L metal in sample} = A \times \frac{(C + B)}{C}$$

where,

A = ug/L of metal in diluted aliquot from calibration curve

B = Acid blank matrix used for dilution, mL

C = sample aliquot, mL

## DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration Determined and resulting PQL
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- Spike Sample Recovery form
- Laboratory Control Sample Summary form
- All Raw Data
- Preparation Records

Client: IDEM-BAA 95-30
SOP ID: SOP-MET-7131-1
Rev. Number: 2.0
Rev. Date: March 1, 1996

# Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Cadmium For Aqueous Samples

Author: Karin Stewart
Prepared For American Analytical, Inc.
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7131

Revision # 2.0
Issued: March 1, 1996

| | |
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| Immediate Supervisor | Date |
| Second Supervisor | Date |
| QA/QC Officer | Date |
| Analyst | Date |

Effective: March 1, 1996

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Client: IDEM-BAA 95-30
SOP ID: SOP-MET-7131-1
Rev. Number: 2.0
Rev. Date: March 1, 1996

# GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF CADMIUM FOR AQUEOUS SAMPLES

## LOCATION:

Metals, Metals Laboratory

## REFERENCE:

SW-846, 3rd Edition, Method 7131

## MATRIX:

Water, Leachates

## QUANTITATION LIMIT:

EQL = 2.0 ug/L; MDL = 0.017 ug/L.

## RANGE:

2.0 ug/L to 20 ug/L without dilution

## PRINCIPLE, SCOPE, AND APPLICATION:

Cadmium in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the

vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

## INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

(1) Successively dilute and reanalyze the samples to eliminate interferences.

(2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-bank absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to $HNO_3$ is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source

of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

## SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during digestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestions are to occur under a fume hood.

## SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous and leachate samples are to be collected in 500 ml plastic containers with teflon lined lids, preserved to pH $\leq$ 2 with nitric acid, and cooled to 4°C until digestion. Samples must be analyzed within 6 months of collection.

## APPARATUS:

1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.

2) Zeeman Graphite Tube Atomizer provides power to fumace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 °C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/min.

3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 ul.

4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and provides data manipulation and reporting of sample calculations.

5) Citizen dot matrix printer, prints calibration and sample results.

6) Class A volumetric pipets

7) Class A volumetric flasks

8) Pipets: Microliter, with disposable tips. sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible sources of contamination prior to their use.

9) Analytical balance

10) Disposable glass serological pipets

ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Furnace optics are cleaned twice weekly. Plumbing connections, and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

REAGENTS AND CALIBRATION STANDARDS:

1) Deionized water - Type II

2) Nitric Acid - concentrated, trace metals grade (Fisher, AS09-212)

3) Furnace stock calibration standard: Using a Class A volumetric pipet, dilute 1.0 ml Cadmium Stock (Spex, QC-19 - 1000 ppm), and 4.0 ml concentrated nitric acid, to 200 ml with DI water in a volumetric flask. Bring to volume. This will result in a final concentration of 10 ppm Cadmium. Dilute stock calibration standard 1:99 with DI water for daily calibration.

4) Furnace ICV/CCV Solutions: Using a 100 uL micropipet and a 10 mL glass serological disposable pipet, transfer 0.10 ml QC-19 Stock (SPEX, QC-19, 100 ppm) and 2.0 ml concentrated nitric acid to a 100 ml Class A volumetric flask partially filled with DI water. Bring to volume. This will result in a 100 ppb final concentration. Dilute 1:1 for a working concentration of 50 ppb.

5) Cadmium Modifier: Using an analytical balance, weigh 1.00 g $NH_4H_2PO_4$ (Fisher, A684-500) and transfer to a 100 mL Class A volumetric flask. Dilute to volume.

CALIBRATION PROCEDURES:

A curve consisting of 4 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of $\geq$ 0.995 to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the PQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCV and CCB must meet the above stated criteria for the ICV and ICB.

SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

## ANALYSIS PROCEDURE:

1.) Turn on monitor, computer, Spectra AA-400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F-6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.

2.) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swap and isopropyl rubbing alcohol. Clean a graphite tube and it's platform using a Kim wipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite tube to the center of the furnace chimney. Swing the toggle lever counter-clockwise in order to close the right-hand housing onto the tube now positioned inside the furnace housing.

3.) Remove rinse bottle and fill to the line with DI $H_2O$. Clean the Blank, Modifier and Standard cups with DI $H_2O$ and 1:1 Nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.

4.) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampling arm will move from it's rinse position to the sample 1 position and than to the sample introduction hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. Use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5.) Open syringe compartment door. Put the syringe clear of it's mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Re-insert the syringe assembly into it's housing and close the compartment door.

6.) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean). The furnace will heat and clean the graphite tube. Press shift and F11 (start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is properly injected onto the platform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it in the path of the UV light and thus putting in view the position of the capillary while inside the graphic tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the analytical signal.

7.) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knobs on the left-hand side of the appropriate lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its

furthest most position from the left-hand baseline.) Note: pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8.) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour check standards into sample cups and place them in their proper positions in the autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for Cadmium:

Cadmium: Program #6, Matrix Modifier - Cadmium Modifier
Standard 1 =0.25 ppb, 2 = 0.50 ppb, 3 = 1.00 ppb, 4 = 2.50 ppb
ICV = 1.00 ppb, CCV = 1.00 ppb

QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within ± 20% of true value.

At least one preparatory blank, laboratory standard, spike and duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See belo

*Method of standard additions:*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

1) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

3) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume $V_x$, are taken. To the first (labeled A) is added a small volume $V_s$ of a standard analyte solution of concentrate $c_s$. To the second (labeled B) is added the same volume $V_s$ of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration $c_x$ is calculated:

$$c_x = \frac{S_B V_S c_S}{(S_A - S_B) V_X}$$

where,

$S_A$ and $S_B$ are the analytical signals (corrected for the blank) of solutions A and B, respectively. $V_S$ and $c_s$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_S$ is made much less than $V_X$, and thus $c_s$ is much greater than $c_x$, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

## DATA TREATMENT:

For determination of metal concentration by direct aspiration and furnace; read the metal valu ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

$$\text{ug/L metal in sample} = A \times \frac{(C + B)}{C}$$

where,

A = ug/L of metal in diluted aliquot from calibration curve

B = Acid blank matrix used for dilution, mL

C = sample aliquot, mL

## DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration Determined and resulting PQL
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- Spike Sample Recovery form
- Laboratory Control Sample Summary form
- All Raw Data
- Preparation Records

**cubs@cubs.com**
10/01/02 10:24 AM

To: Alan Baumann/R5/USEPA/US@EPA
cc:
Subject: Come Join Us At Wrigley Field

Dear Wrigleyville Neighbor:

Come Join Us At Wrigley Field!

Thank you for supporting our proposed improvements to Wrigley Field. We've received thousands of positive responses from our neighbors and greatly appreciate your support as we continue to work with neighborhood groups and the City of Chicago to finalize our plans for improvements and additional night games.

To express our thanks for your interest and support, we're inviting you to join us and other members of Wrigleyville Neighbors for lunch at Wrigley Field between 11:00 a.m. and 1:00 p.m. on Saturday, October 5.

We'll provide the food and refreshments and you'll be able to run the bases, play catch and take pictures in front of the ivy-covered wall.

Please RSVP by 2:00 p.m. on Friday by calling 888-871-CUBS (2827). For security reasons (and to ensure we have enough food), only those who RSVP will be allowed in. IMPORTANT: You must bring a photo ID with you to the ball park.

If you know someone in the neighborhood who wants to join Wrigleyville Neighbors, please have them RSVP so we can sign them up on Saturday.

Hope to see you there!

Andy MacPhail
President

Mark E. McGuire
Executive Vice President, Business Operations

# DOCUMENTS MANAGEMENT SECTION



**Timeframe report for Fiscal Year 2002**
**All Assignees**

| | 1-10 | 11-20 | 21-30 | 31-40 | 41-50 | 51-60 | 61-70 | 71-80 | 81-90 | 91-100 | >100 | Open |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| October | 25 | 64 | 12 | 6 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| November | 28 | 65 | 4 | 5 | 0 | 3 | 2 | 0 | 0 | 0 | 0 | 0 |
| December | 19 | 36 | 5 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Quarter 1 | 72 | 165 | 21 | 13 | 1 | 4 | 2 | 0 | 0 | 0 | 0 | 0 |
| January | 29 | 40 | 3 | 3 | 3 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| February | 30 | 65 | 5 | 3 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 |
| March | 34 | 57 | 3 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Quarter 2 | 93 | 162 | 11 | 6 | 4 | 1 | 0 | 1 | 0 | 0 | 0 | 0 |
| April | 22 | 67 | 8 | 2 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| May | 18 | 70 | 8 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| June | 16 | 75 | 6 | 4 | 0 | 2 | 0 | 0 | 0 | 0 | 0 | 0 |
| Quarter 3 | 56 | 212 | 22 | 7 | 2 | 3 | 0 | 0 | 0 | 0 | 0 | 0 |
| July | 17 | 57 | 3 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| August | 23 | 65 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 5 |
| September | 9 | 5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 63 |
| Quarter 4 | 49 | 127 | 6 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 68 |
| Report Total | 270 | 666 | 60 | 28 | 7 | 8 | 2 | 1 | 0 | 0 | 0 | 68 |

# STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL OR DISSOLVED METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA OR FLAME ATOMIC ABSORPTION SPECTROSCOPY

Originating Author: Karin Stewart
Revision Author: Troy Goehl

This SOP is effective upon signed approval by the following:

Troy Goehl — Unit Supervisor — 12-13-2001 — Date

[signature] — QA/QC Director — 12-13-2001 — Date

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

# 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This procedure is based EPA Method 200.7 and SW-846 Method 3010A. This procedure is applicable to the digestion of all aqueous samples and the extracts from the TCLP and SPLP procedures. This procedure is not applicable for the preparation of samples to be analyzed by Graphite Furnace Atomic Absorption Spectroscopy.

## 3.0 SUMMARY

3.1 1.5 ml of concentrated nitric acid is added to a 50 ml aliquot of sample. The mixture is heated near boiling. An additional 2 ml of nitric acid is added to the sample and the mixture is again heated to near boiling. 5 ml of a 50% hydrochloric acid solution is added to the mixture and the mixture gently heated, cooled and diluted to a final volume of 50 ml with DI water.

3.2 This procedure is a combination of the EPA methods referenced in section 17.0. There are subtle differences in the acid volumes used in the reference methods. This procedure uses a 50 ml aliquot of sample, whereas the reference methods are based on 100 ml. The volumes of acid used have been adjusted accordingly and, where different volumes of acid are listed in the reference methods, the average of the two is used. These changes from the written methods are considered acceptable as shown through the continued generation of acceptable control samples and performance evaluation samples.

## 4.0 DEFINITIONS

4.1 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.2 Analyte – The specific component measured in a chemical analysis.

4.3 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Calibration Blank – An aliquot of the standard diluent (water or organic solvent) that is not carried through the sample preparation scheme. It is analyzed to verify that the analytical system is free from contamination. Also referred to as an instrument blank or solvent blank.

- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.

- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination

in sample preparation and analyses. Also referred to as a Preparation or Procedural Blank.

4.4 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.5 Laboratory Control Sample (LCS) – An aliquot of clean matrix (lab pure water or vendor supplied solid) spiked with target analytes or compounds representative of target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.6 Laboratory Control Sample Duplicate (LCSD) – An aliquot of laboratory pure reagent spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.7 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.8 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.9 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.10 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.11 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.12 Sample – A portion of material supplied by the client for analysis.

4.13 Sample Duplicate – Two aliquots of the same sample processed independently. This monitors precision of the analysis. Precision results are reported as relative percent difference (RPD).

## 5.0 INTERFERENCES

5.1 The digestion procedure may not be sufficient to completely break down some metal complexes. Additional digestion time or a more vigorous digestion may be required to facilitate complete digestion. Typically, complete oxidation is evidenced by a light color or no change upon addition of acid or continued heating.

5.2 Precipitation during digestion may cause a suppression of the total Silver content measured in the analysis. If this occurs, dilute the sample prior to digestion to lower the effective Silver concentration to below 1 ppm.

5.3 Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Labware suspected of causing contamination should be soaked with 1:5 nitric acid and rinsed thoroughly with lab pure water.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Digestion vessels, Environmental Express catalog #SC475 or equivalent

7.3 Filters, Environmental Express FilterMate, catalog #SC0401, or equivalent

7.4 Ribbed watch glasses, Environmental Express catalog #SC505 or equivalent

7.5 100 ml graduated cylinders

7.6 Digestion block capable of maintaining 90 to 95°C

7.7 Class A volumetric pipets

7.8 Oxford-style repipetters

7.9 Vacuum filtration device

7.10 0.45 micron filters

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

8.2.1 Lab pure water. ASTM Type II water is generated in accordance with the procedure described in the Quality Assurance Plan.

8.2.2 Nitric acid, conc. $HNO_3$: Trace metals grade (Fisher, AS09-212 or equivalent)

8.2.3 Hydrochloric acid, conc. HCl: Trace metals grade (Fisher, AS08-212 or equivalent)

8.3 Standards

8.3.1 Stock ICP Spike Standard 1: Inorganic Ventures catalog #SIMA-SPIKE-1, or equivalent, contains the following constituents. Store this standard in the standards cabinet located in the metals instrument lab.

| ELEMENT | CONC., ug/ml |
|---|---|
| Ca, Mg, Na | 10,000 |
| Al, Pb, Ni, Tl, V, Zn | 1000 |
| Ba, Be, Cd, Co, Cu, Mn, Ag, Sr | 100 |

8.3.2 Working ICP Spike Standard 1: In a 250 ml volumetric flask, dilute 25 ml of the stock ICP spike standard 1 to the mark with 12.5 ml conc. HCl, 5 ml conc. $HNO_3$ and DI water. This prepares a standard of the following concentrations. Store this standard in the standards cabinet located in the metals instrument lab.

| ELEMENT | CONC., ug/ml |
|---|---|
| Ca, Mg, Na | 1000 |
| Al, Pb, Ni, Tl, V, Zn | 100 |
| Ba, Be, Cd, Co, Cu, Mn, Ag, Sr | 10 |

8.3.3 Stock ICP Spike Standard 2: Inorganic Ventures catalog #SIMA-SPIKE-2, or equivalent, contains the following constituents. Store this standard in the standards cabinet located in the metals instrument lab.

| ELEMENT | CONC., ug/ml |
|---|---|
| K | 10,000 |
| Sb, As, B, Cr, Fe, Mo, P, Se, Si, S, Sn, Ti | 1000 |

8.3.4 Working ICP Spike Standard 2: In a 250 ml volumetric flask, dilute 25 ml of the stock ICP spike standard 2 to the mark with 12.5 ml conc. HCl, 5 ml conc. $HNO_3$ and DI water. This prepares a standard of the following concentrations. Store this standard in the standards cabinet located in the metals instrument lab.

| ELEMENT | CONC., ug/ml |
|---|---|
| K | 1000 |
| Sb, As, B, Cr, Fe, Mo, P, Se, Si, S, Sn, Ti | 100 |

8.3.5 LCS/MS: Add 1 ml of each of the working ICP spike solutions to 50 ml DI water or sample to prepare the LCS or MS, respectively. This prepares a control sample with the following constituents.

| Aqueous LCS/MS | Conc., mg/l | Aqueous LCS/MS | Conc., mg/l |
|---|---|---|---|
| Aluminum | 2.0 | Manganese | 0.2 |
| Antimony | 2.0 | Molybdenum | 2.0 |
| Arsenic | 2.0 | Nickel | 2.0 |
| Barium | 0.2 | Potassium | 20 |
| Beryllium | 0.2 | Selenium | 2.0 |
| Boron | 2.0 | Silicon | 2.0 |
| Cadmium | 0.2 | Silver | 0.2 |
| Calcium | 20 | Sodium | 20 |
| Chromium | 2.0 | Strontium | 0.2 |
| Cobalt | 0.2 | Thallium | 2.0 |
| Copper | 0.2 | Tin | 2.0 |
| Iron | 2.0 | Titanium | 2.0 |
| Lead | 2.0 | Vanadium | 2.0 |
| Magnesium | 20 | Zinc | 2.0 |

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Samples should be collected in a plastic container. For Total Metals, preservation consists of $HNO_3$ to pH < 2. If samples are preserved in the lab, the samples must set for 16 hours prior to pH verification and subsampling. For Dissolved Metals, the sample must be filtered through a 0.45-micron filter prior to acidification.

9.3 Digestion must be performed within the maximum allowable hold time of 180 days from collection.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed by each analyst prior to unsupervised sample preparation and whenever substantial change has occurred in the procedure. Prepare four separate Laboratory Control Standards. These standards must be from a source different from that used for instrument calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.2 A *Method Blank* must be prepared with each batch of maximum 20 samples and at a minimum of one per day. Also, the TCLP/SPLP method blanks must be prepared using the extraction fluid used in the given samples.

10.3 A *Laboratory Control Sample* must be prepared with each batch of maximum 20 samples and at a minimum of one per day. Also, the TCLP/SPLP LCS must be prepared using the extraction fluid used in the given samples.

10.4 A *Matrix Spike and Matrix Spike Duplicate* must be prepared with each batch of maximum 20 samples and at a minimum of one per day. If insufficient sample is available for the preparation of the MS/MSD, a duplicate LCS (LCSD) or a MS on two separate samples must be prepared.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Repipetters must be verified on a weekly basis. Details of this requirement and the procedure used to verify their operation are in the Calibration of Manual Repipetters SOP.

11.2 Verify the operation of the digestion block. Temperature must be maintained in the range of 90 – 95°C.

## 12.0 PROCEDURE

12.1 Prior to use, rinse digestion vessels, graduated cylinders, funnels, filtration device, and watch glasses with 1:1 nitric acid and 3 rinses of DI water.

12.2 Shake sample vigorously and transfer 50 ml of sample directly into a digestion vessel. For dissolved metals filter an adequate volume of sample using the vacuum filtration device unless sample was filtered in the field.

12.3 Using an Oxford Macro pipet under a fume hood, add 1.5 ml of concentrated $HNO_3$ to the sample and swirl to mix.

12.4 Place the digestion vessel in the digestion block for two hours. Do not allow the digestate to boil. Do not allow the vessel to go to dryness. If the vessel goes to dryness, the digestion must be started again with a new aliquot of sample.

12.5 Cool the digestate to room temperature.

12.6 Add 2 ml of concentrated HNO3 to the sample and swirl to mix.

12.7 Place the digestion vessel in the digestion block for 30 minutes. If oxidation is not complete after this second addition of acid and the heating process, add additional $HNO_3$ in 1 ml increments until complete. (Complete oxidation can be visually evaluated. The production of a light colored digestate or no change in appearance with continued digestion is typically indicative of complete oxidation.)

12.8 Add 2.5 ml of DI water and 2.5 ml conc. HCl to the sample, in that order, and swirl to mix.

12.9 Place in digestion block for 15 minutes.

12.10 Cool the digestate to room temperature.

12.11 Dilute, in the digestion vessel, the sample volume to 50 ml with DI water and filter if particulate matter is present.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 Sample preparation is documented on the Metals Digestion Log Sheet.

13.2 Enter the preparation data in the LIMS. Details on the procedure for entering analytical data are in the Preparation Batch Data Entry SOP.

## 14.0 METHOD PERFORMANCE

Not applicable.

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Refer to the SIMALABS International Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

## 17.0 REFERENCES

17.1 USEPA Method 200.7, December 1982

17.2 SW-846 Method 3010A

17.3 SIMALABS International Quality Assurance Plan, current revision

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

None

# STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF AQUEOUS SAMPLES USING LIQUID-LIQUID EXTRACTION BY SW-846 METHOD 3510C

Originating Author: Karin Stewart
Revision Author: Jeff Loewe

This SOP is effective upon signed approval by the following:

Unit Supervisor — Date: 6/26/01

QA/QC Director — Date: 6-20-2001

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

# 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This is an extraction procedure for the preparation of samples for Pesticide, Polychlorinated Biphenyl, Polyaromatic Hydrocarbon, and Semi-Volatile Organic Analytes. This procedure is applicable to the preparation of aqueous and non-aqueous liquid matrix samples.

## 3.0 SUMMARY

3.1 This process involves the isolation and concentration of organic compounds from aqueous samples for a variety of chromatographic techniques.

3.2 A measured volume of sample, usually 1 liter, at a specified pH, is serially extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or analytical method used.

## 4.0 DEFINITIONS

4.1 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.2 Analyte – The specific component measured in a chemical analysis.

4.3 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.

- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Method Blank.

4.4 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.5 Laboratory Control Sample (LCS) – An aliquot of laboratory pure reagent spiked with target analytes or compounds representative of target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.6 Laboratory Control Sample Duplicate (LCSD) – An aliquot of laboratory pure reagent spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the

two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.7 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.8 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.9 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.10 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.11 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.12 Sample – A portion of material supplied by the client for analysis.

4.13 Sample Duplicate – Two aliquots of the same sample processed independently. This monitors precision of the analysis. Precision results are reported as relative percent difference (RPD).

4.14 Solvent exchange – Adding a different solvent other than the original extraction solvent and evaporating off the original solvent.

## 5.0 INTERFERENCES

5.1 Interferences that co-elute vary considerably from sample to sample.

5.2 If the analysis of an extracted sample is prevented due to matrix interferences, further clean-up of the extract may be required.

5.3 Phthalate esters can contaminate many types of plasticware and glassware products used in the lab. Plastics, in particular, must be avoided because phthalates are commonly used in plasticizers and are easily extracted from plastic

materials. Phthalate contamination may easily result any time that consistent adherence to the quality control requirements are not practiced.

5.4 Soap residue may cause the degradation of certain analytes especially aldrin, heptachlor, and most organophosphorus pesticides. Strict adherence to the Glassware Washing SOP is required.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Turbo Vap II concentrator (water bath = 33°C) and tubes

7.3 2L Teflon separatory funnels

7.4 Glass funnels

7.5 Glass wool

7.6 Disposable pipettes, 1 and 10 ml

7.7 Graduated cylinders, glass, 100 and 1000 ml

7.8 2 ml autosampler vials and caps

7.9 Test tubes with caps

7.10 VOA vials

7.11 Centrifuge

7.12 pH paper

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook.

8.2 Reagents

8.2.1 Lab pure water

8.2.2 Acetone ($C_3H_6O$)

8.2.3 Acetonitrile ($C_2H_3N$)

8.2.4 Hexane ($C_6H_{14}$)

8.2.5 Methanol ($CH_4O$, also noted as MeOH)

8.2.6 Methylene chloride ($CH_2Cl_2$)

8.2.7 Sodium sulfate ($Na_2SO_4$)

8.2.8 Sulfuric acid, concentrated $H_2SO_4$

8.2.9 Sodium hydroxide (NaOH)

8.2.10 Sodium hydroxide, 10N NaOH: In a 1L volumetric flask, dissolve and dilute 400 g NaOH to the mark with DI water.

8.3 Standards

8.3.1 Stock Base-Neutral Spike Standard, 1000 ug/ml each: Supelco #502294. See table in section 18.0 for compound list.

8.3.2 Stock Acid Spike Standard, 2000 ug/ml each: Supelco #502308. See table in section 18.0 for compound list.

8.3.3 SVOA Spike: In a 50 ml volumetric flask, dilute 2.5 ml of the stock base-neutral spike standard and 2.5 ml of the stock acid spike standard to the mark with MeOH. This prepares a standard containing the base-neutral compounds at 50 ug/ml and the acid compounds at 100 ug/ml. **Add 1 ml of this solution to the LCS, MS, and MSD samples.**

8.3.4 Stock Base-Neutral Surrogate Standard, 5000 ug/ml each: Supelco #4-7262. See table in section 18.0 for compound list.

8.3.5 Stock Acid Surrogate Standard, 10,000 ug/ml each: Supelco #4-7261. See table in section 18.0 for compound list.

8.3.6 SVOA Surrogate Standard: In a 100 ml volumetric flask, dilute 2.0 ml of the stock base-neutral surrogate standard and 1.5 ml of the stock acid surrogate standard to the mark with MeOH. This prepares a standard containing the base-neutral compounds at 100 ug/ml and the acid compounds at 150 ug/ml. **Add 1 ml of this solution to all samples.**

8.3.7 PNA-IL Surrogate Standard, 10 ug/ml each: Ina 200 ml volumetric flask, dilute 500 ul of the stock base-neutral surrogate standard to the mark with MeOH. **Add 1 ml of this solution to all samples.**

8.3.8 Stock PNA-IL Spike Standard, 2000 ug/ml each: Accustandard #Z-014G-R-PAK.

8.3.9 PNA-IL Spike Standard, 10 ug/ml each: In a 100 ml volumetric flask, dilute 500 ul of the stock PNA-IL spike standard to the mark with MeOH. **Add 1 ml of this solution to the LCS, MS, and MSD samples.**

8.3.10 Stock Phenol Surrogate Standard, 2000 ug/ml each: Accustandard #M-8040-SS-PAK contains 2-Fluorophenol and 2,4,6-Tribromophenol.

8.3.11 Phenol Surrogate Standard, 100 ug/ml: In a 25 ml volumetric flask, dilute 1.25 ml of the stock phenol surrogate standard to the mark with acetone. **Add 1 ml of this solution to all samples.**

8.3.12 Stock HPLC PNA Surrogate Standard, 2000 ug/ml: Accustandard #M-625-04-10X contains Decachlorobiphenyl (DCB).

8.3.13 HPLC PNA Surrogate Standard, 50 ug/ml: In a 50 ml volumetric flask, dilute 1.25 ml of the stock HPLC PNA surrogate standard to the mark with acetonitrile. **Add 1 ml of this solution to all samples.**

8.3.14 Stock PCB Spike Standard, 1000 ug/ml: Supelco #4-4809 contains Aroclor 1260.

8.3.15 PCB Spike Standard, 5 ug/ml: In a 100 ml volumetric flask, dilute 500 ul of the stock PCB spike standard to the mark with hexane. **Add 1 ml of this solution to the LCS, MS, and MSD.**

8.3.16 Stock Pesticide Spike Standard, 2000 ug/ml each: Supelco #4-8913. See the table in section 18.0 for the compound list.

8.3.17 Pesticide Spike Standard, 0.5 ug/ml each: In a 100 ml volumetric flask, dilute 25 ul of the stock pesticide spike standard to the mark with acetone. **Add 1 ml of this solution to the LCS, MS, and MSD.**

8.3.18 Stock Pest/PCB Surrogate Standard, 200 ug/ml each: Accustandard #CLP-032-K contains DCB and TCMX.

8.3.19 Pest/PCB Surrogate Standard, 0.2 ug/ml each: In a 200 ml volumetric flask, dilute 200 ul of the stock pest/PCB surrogate standard to the mark with hexane. **Add 1 ml of this solution to all samples.**

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Samples should be collected in a 1L amber glass container. Preservation consists of storage in the range of 0.1-6°C.

9.3 Preparation must be performed within the maximum allowable hold time of 7 days from collection.

## 10.0 QUALITY CONTROL

10.1 A *Laboratory Control Standard* must be extracted with each batch of maximum 20 samples and at a minimum of one per day analyzed.

10.2 A *Method Blank* must be extracted with each LCS.

10.3 A *Matrix Spike and Matrix Spike Duplicate* sample must be extracted with each group of maximum 10 samples at a minimum of one per day extracted for the 600 series methods with the exception of method 625, and with each group of maximum 20 samples at a minimum of one per day extracted for the 8000 series methods and method 625. If insufficient sample exists for the preparation of a MS/MSD, a duplicate LCS (i.e. LCSD) should be extracted.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Perform the required preventative maintenance as necessary.

11.2 Check water level in the Turbo Vap II. Add DI water as needed.

11.3 Check temperature of the Turbo Vap II water bath and adjust as needed.

## 12.0 PROCEDURE

12.1 Rinse all glassware with acetone.

12.2 Triple rinse all glassware with methylene chloride.

12.3 Transfer sample into separatory funnel. The MB and LCS are prepared with DI water adjusted to the proper pH.

12.4 If necessary, adjust pH using sulfuric acid or sodium hydroxide. See table in section 18.0 for the required sample pH.

12.5 Add surrogate and spike solutions to appropriate samples and record type, lot number, and amount added.

12.6 Add 60 ml methylene chloride to separatory funnel.

12.7 Shake separatory funnel for 2 minutes, venting frequently.

12.8 Prepare funnel filter with glass wool and sodium sulfate.

12.9 Rinse filter with approximately 20 ml of methylene chloride and discard the methylene chloride.

12.10 Drain extract from separatory funnel through filter into a concentrator tube. (Occasionally samples will need to be centrifuged to complete the separation).

12.11 Repeat steps 12.6, 12.7, and 12.10 two more times.

12.12 Rinse filter with methylene chloride adding this to the concentrator tube.

12.13 Place concentrator tube into Turbo Vap II.

12.14 If needed, "solvent exchange" the extract when its volume is below 1 ml and continue to evaporate extract to below 1 ml then remove from the Turbo Vap II. If extract does not need a solvent exchange, simply remove extract from Turbo Vap II when extract falls below 1 ml. **CAUTION: DO NOT LET EXTRACT GO DRY!** See table in section 18.0 for the required final solvent.

If a secondary fraction is to be extracted, adjust pH of sample using sulfuric acid or sodium hydroxide. Then repeat steps 12.6, 12.7 and 12.10, and combine the two fractions in the same concentrator tube.

12.15 Using a 1 ml syringe, measure and remove extract from concentrator tube and place in appropriate container (e.g. vials, test tubes, etc.) labeled with the sample I.D., fraction, volume, parameter, and extraction personnel initials.

12.16 Rinse concentrator tube walls with 2-3 ml of appropriate solvent.

12.17 Using rinse solvent in concentrator tube, adjust volume of extract appropriate volume.

12.18 Cap container of extract.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 Enter sample preparation data into the LIMS system.

## 14.0 METHOD PERFORMANCE

14.1 Not applicable.

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Dispose of any resulting residue, digestate, or extract in accordance with local sanitary regulations.

16.2 Additional sample shall be disposed of properly following the completion of analysis and an appropriate additional holding time.

## 17.0 REFERENCES

17.1 SW-846 Method 3510C

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

| BN Spike Standard Compounds | |
|---|---|
| Acenapthene | 1,2,4-Trichlorobenzene |
| N-Nitrosodi-N-propylamine | 1,3-Dichlorobenzene |
| Pyrene | 2,4-Dinitrotoluene |

| Acid Spike Standard Compounds | |
|---|---|
| Pentachlorophenol | 4-Chloro-3-methylphenol |
| Phenol | 4-Nitrophenol |
| 2-Chlorophenol | |

| BN Surrogate Standard Compounds | |
|---|---|
| Nitrobenzene-d5 | 1,2-Dichlorobenzene-d4 |
| p-Terphenyl-d14 | 2-Fluorobiphenyl |

| Acid Surrogate Standard Compounds | |
|---|---|
| Phenol-d6 | 2,4,6-Tribromophenol |
| 2-Chlorophenol-d4 | 2-Fluorophenol |

| PNA Spike Standard Compounds | |
|---|---|
| Naphthalene | Benzo(b)fluoranthene |
| Acenaphthene | Benzo(k)fluoranthene |
| Acenaphthylene | Benzo(a)pyrene |
| Flourene | Dibenzo(a,h)anthracene |
| Pyrene | Benzo(g,h,i)perylene |
| Benzo(a)anthracene | Indeno(1,2,3-cd)pyrene |
| Chrysene | |

| Pesticide Spike Standard Compounds | |
|---|---|
| Aldrin | Endrin aldehyde |
| Alpha-BHC | Endrin ketone |
| Beta-BHC | Gamma-BHC |
| Delta-BHC | Heptachlor |
| Dieldrin | Heptachlor epoxide |
| Endosulfan I | Methoxychlor |
| Endosulfan II | 4,4'-DDD |
| Endosulfan sulfate | 4,4'-DDE |
| Endrin | 4,4'-DDT |

| Extraction Conditions | | | | | | |
|---|---|---|---|---|---|---|
| Determinative Method and Prep Code | Analyte Group | Initial Extraction pH | Secondary Extraction pH | Final Solvent for Analysis | Final Solvent for Cleanup | Final Vol., ml |
| 8041<br>3510_Phenol | Phenols | ≤ 2 | None | None | Hexane | 1 |
| 8081A<br>3510_Pest | Pesticides | 5 – 9 | None | Hexane | Hexane | 10 |
| 8082<br>3510_PCB | PCBs | 5 – 9 | None | Hexane | Hexane | 10 |
| 8270C[1]<br>3510_B | SVOA (BNA) | <2 | >11 | None | --- | 1 |
| 8310<br>3510_HPLC | PAH (PNA) | As received | None | Acetonitrile | --- | 1 |

1 = Extraction pH sequence may be reversed to better separate the acid and neutral components. Excessive pH adjustments may result in the loss of some analytes.

# STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF NON-AQUEOUS SAMPLES FOR TOTAL METALS ANALYSIS

Originating Author: Karin Stewart
Revision Author: Troy Goehl

This SOP is effective upon signed approval by the following:

Troy Goehl
Unit Supervisor

12-13-2001
Date

QA/QC Director

12-13-2001
Date

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

# 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This procedure is based SW-846 Method 3050B. This procedure is applicable to the digestion of all non-aqueous samples. Separate procedural steps are included depending on the instrument used for analysis.

## 3.0 SUMMARY

3.1 10 ml of 1:1 nitric acid is added to approximately 1g sample. The mixture is heated for 15 minutes then allowed to cool. 5 ml of concentrated nitric acid is added to the sample and the mixture is again heated to near boiling for 2 hours. DI water and hydrogen peroxide are then added and the mixture heated for an additional 2 hours. The digestate is then diluted to volume for analysis by GFAA. Analysis by ICP or FLAA requires the addition of 10 ml concentrated hydrochloric acid and additional heating prior to final dilution to 100 ml with DI water.

3.2 This procedure produces 50 ml of digestate, whereas the reference method results in 100 ml. The volumes of acid and hydrogen peroxide used have been adjusted accordingly. These changes from the written methods are considered acceptable as shown through the continued generation of acceptable control samples and performance evaluation samples.

## 4.0 DEFINITIONS

4.1 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.2 Analyte – The specific component measured in a chemical analysis.

4.3 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Calibration Blank – An aliquot of the standard diluent (water or organic solvent) that is not carried through the sample preparation scheme. It is analyzed to verify that the analytical system is free from contamination. Also referred to as an instrument blank or solvent blank.

- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.

- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Preparation or Procedural Blank.

4.4 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.5 Laboratory Control Sample (LCS) – An aliquot of clean matrix (lab pure water or vendor supplied solid) spiked with target analytes or compounds representative of target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.6 Laboratory Control Sample Duplicate (LCSD) – An aliquot of laboratory pure reagent spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.7 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.8 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.9 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.10 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.11 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.12 Sample – A portion of material supplied by the client for analysis.

4.13 Sample Duplicate – Two aliquots of the same sample processed independently. This monitors precision of the analysis. Precision results are reported as relative percent difference (RPD).

## 5.0 INTERFERENCES

5.1 The digestion procedure may not be sufficient to completely break down some metal complexes. Additional digestion time or a more vigorous digestion may be required to facilitate complete digestion. Typically, complete oxidation is evidenced by a light color or no change upon addition of acid or continued heating.

5.2 Precipitation during digestion may cause a suppression of the total Silver content measured in the analysis. If this occurs, dilute the sample prior to digestion to lower the effective Silver concentration to below 1 ppm.

5.3 Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Labware suspected of causing contamination should be soaked with 1:5 nitric acid and rinsed thoroughly with lab pure water.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Digestion vessels, Environmental Express catalog #SC475 or equivalent

7.3 Filters, Environmental Express FilterMate, catalog #SC0401, or equivalent

7.4 Ribbed watch glasses, Environmental Express catalog #SC505 or equivalent

7.5 100 ml graduated cylinders

7.6 Digestion block capable of maintaining 90 to 95°C

7.7 Class A volumetric pipets

7.8 Oxford-style repipetters

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

8.2.1 Lab pure water. ASTM Type II water is generated in accordance with the procedure described in the Quality Assurance Plan.

8.2.2 Nitric acid, conc. $HNO_3$: Trace metals grade (Fisher, AS09-212 or equivalent)

8.2.3 Nitric acid, 1:1 $HNO_3$: Dilute 1 volume of conc. $HNO_3$ with an equal volume of DI water.

8.2.4 Hydrochloric acid, conc. HCl: Trace metals grade (Fisher, AS08-212 or equivalent)

8.2.5 Hydrogen Peroxide, 30% $H_2O_2$: Fisher, H325-500 or equivalent

8.3 Standards

8.3.1 Stock ICP Spike Standard 1: Inorganic Ventures catalog #SIMA-SPIKE-1, or equivalent, contains the following constituents. Store this standard in the standards cabinet located in the metals instrument lab.

| ELEMENT | CONC., ug/ml |
|---|---|
| Ca, Mg, Na | 10,000 |
| Al, Pb, Ni, Tl, V, Zn | 1000 |
| Ba, Be, Cd, Co, Cu, Mn, Ag, Sr | 100 |

8.3.2 Working ICP Spike Standard 1: In a 250 ml volumetric flask, dilute 25 ml of the stock ICP spike standard 1 to the mark with 12.5 ml conc. HCl, 5 ml conc. $HNO_3$ and DI water. This prepares a standard of the following concentrations. Store this standard in the standards cabinet located in the metals instrument lab.

| ELEMENT | CONC., ug/ml |
|---|---|
| Ca, Mg, Na | 1000 |
| Al, Pb, Ni, Tl, V, Zn | 100 |
| Ba, Be, Cd, Co, Cu, Mn, Ag, Sr | 10 |

8.3.3 Stock ICP Spike Standard 2: Inorganic Ventures catalog #SIMA-SPIKE-2, or equivalent, contains the following constituents. Store this standard in the standards cabinet located in the metals instrument lab.

| ELEMENT | CONC., ug/ml |
|---|---|
| K | 10,000 |
| Sb, As, B, Cr, Fe, Mo, P, Se, Si, S, Sn, Ti | 1000 |

8.3.4 Working ICP Spike Standard 2: In a 250 ml volumetric flask, dilute 25 ml of the stock ICP spike standard 2 to the mark with 12.5 ml conc. HCl, 5 ml conc. $HNO_3$ and DI water. This prepares a standard of the following concentrations. Store this standard in the standards cabinet located in the metals instrument lab.

| ELEMENT | CONC., ug/ml |
|---|---|
| K | 1000 |
| Sb, As, B, Cr, Fe, Mo, P, Se, Si, S, Sn, Ti | 100 |

8.3.5 MS: Add 1 ml of each of the working ICP spike solutions to a measured amount of sample to prepare the MS. This prepares a control sample with the following constituents.

| Non-aqueous MS | Conc., mg/l | Non-aqueous MS | Conc., mg/l |
|---|---|---|---|
| Aluminum | 2.0 | Manganese | 0.2 |
| Antimony | 2.0 | Molybdenum | 2.0 |
| Arsenic | 2.0 | Nickel | 2.0 |
| Barium | 0.2 | Potassium | 20 |
| Beryllium | 0.2 | Selenium | 2.0 |
| Boron | 2.0 | Silicon | 2.0 |
| Cadmium | 0.2 | Silver | 0.2 |
| Calcium | 20 | Sodium | 20 |
| Chromium | 2.0 | Strontium | 0.2 |
| Cobalt | 0.2 | Thallium | 2.0 |
| Copper | 0.2 | Tin | 2.0 |
| Iron | 2.0 | Titanium | 2.0 |
| Lead | 2.0 | Vanadium | 2.0 |
| Magnesium | 20 | Zinc | 2.0 |

8.3.6 LCS: Environmental Resource Associates catalog #540 contains 28 elements at various concentrations. The certified values and acceptance criteria must be updated in the LIMS when a new lot number is used. Prepare this control sample in the same fashion as environmental samples, using a sample size appropriate for the concentrations in the standard (typically, between 0.25 and 1g of standard is appropriate). Store this standard in the metals sample preparation area.

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Thermal preservation consists of storage of the sample in the range of 0.1 – 6°C. Samples are stored in the main walk-in cooler.

9.3 Digestion must be performed within the maximum allowable hold time of 180 days from collection.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed by each analyst prior to unsupervised sample preparation and whenever substantial change has occurred in the procedure. Prepare four separate Laboratory Control Standards. These standards must be from a source different from that used for instrument calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.2 A *Method Blank* must be prepared with each batch of maximum 20 samples and at a minimum of one per day.

10.3 A *Laboratory Control Sample* must be prepared with each batch of maximum 20 samples and at a minimum of one per day.

10.4 A *Matrix Spike and Matrix Spike Duplicate* must be prepared with each batch of maximum 20 samples and at a minimum of one per day. If insufficient sample is available for the preparation of the MS/MSD, a duplicate LCS (LCSD) or a MS on two separate samples must be prepared.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Repipetters must be verified on a weekly basis. Details of this requirement and the procedure used to verify their operation are in the Calibration of Manual Repipetters SOP.

11.2 Verify the operation of the digestion block. Temperature must be maintained in the range of 90 – 100°C.

## 12.0 PROCEDURE

12.1 Prior to use, rinse digestion vessels, graduated cylinders, funnels, filtration device, and watch glasses with 1:1 nitric acid and 3 rinses of DI water.

12.2 Thoroughly mix and transfer approximately 1g of sample directly into a digestion vessel.

12.3 Using an Oxford Macro pipet under a fume hood, add 2.5 ml DI water and 2.5 ml conc. $HNO_3$ to the sample, in that order, and swirl to mix.

12.4 Cover vessel with a ribbed watch glass and place in the digestion block for 15 minutes.

12.5 Cool the digestate to room temperature.

12.6 Add 2.5 ml conc. $HNO_3$, cover the sample with a ribbed watch glass, and place in the digestion block for 30 minutes. If a brown vapor appears within the 30 minutes, this step must be repeated. If no brown vapor exists, allow the sample to digest for a minimum of one hour to a maximum of two hours. Do not allow the digestate to boil. Do not allow the vessel to go to dryness. (Add a small amount of water if necessary.) If the vessel goes to dryness, the digestion must be started again with a new aliquot of sample.

12.7 Cool the digestate to room temperature.

12.8 Add 1 ml DI water and 1.5 ml 30% $H_2O_2$, cover the sample with a ribbed watch glass, and place in the digestion block until no further effervescence is apparent.

12.9 Repeat the additions of 0.5 ml $H_2O_2$ followed by the heating until no further effervescence occurs or until a maximum of 5 ml $H_2O_2$ is added. When the addition of $H_2O_2$ no longer causes effervescence, continue heating for a minimum of one hour to a maximum of two hours. Do not allow the digestate to boil. Do not allow the vessel to go to dryness. (Add a small amount of water if necessary.)

12.10 Follow the next step(s) as appropriate depending on the method of analysis.

12.10.1 (For GFAA analysis only) Allow the sample to cool to room temperature, dilute to 50 ml with DI water and filter the particulate matter.

12.10.2 (For ICP or FLAA analysis only) Add 5 ml conc. HCl, cover with a ribbed watch glass, and place in the digestion block for 15 minutes.

12.10.2.1 Cool the digestate to room temperature.

12.10.2.2 Dilute the sample volume to 50 ml with DI water and filter the particulate matter.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 Sample preparation is documented on the Metals Digestion Log Sheet.

13.2 Enter the preparation data in the LIMS. Details on the procedure for entering analytical data are in the Preparation Batch Data Entry SOP.

## 14.0 METHOD PERFORMANCE

Not applicable.

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Refer to the SIMALABS International Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

## 17.0 REFERENCES

17.1 SW-846 Method 3050B

17.2 SIMALABS International Quality Assurance Plan, current revision

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

None

# STANDARD OPERATING PROCEDURE FOR METALS BY INDUCTIVELY COUPLED PLASMA EMISSION SPECTROMETRY USING EPA METHOD 200.7 AND SW-846 METHOD 6010B

Originating Author: Unknown
Revision Author: Troy Goehl

This SOP is effective upon signed approval by the following:

Troy Goehl
Unit Supervisor — Date: 12/14/2001

Mary McFarlane
QA/QC Director — Date: 12-14-2001

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

# 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This is an Inductively Coupled Plasma – Atomic Emission Spectrometry procedure for the determination of various metal elements. This procedure is applicable to the analysis of digestates from aqueous, non-aqueous liquid, drinking water, and solid matrix samples. The applicable elements, MDLs and routine reporting limits (PQL) are listed in the table below. Lower PQLs may be reported upon client request. The "low level" test code is used to indicate this request.

| Analyte | MDL mg/l | PQL mg/l |
|---|---|---|
| Aluminum | 0.023 | 0.2 |
| Antimony | 0.028 | 0.1 |
| Arsenic | 0.026 | 0.1 |
| Barium | 0.002 | 0.01 |
| Beryllium | 0.0004 | 0.01 |
| Boron | 0.009 | 0.1 |
| Cadmium | 0.002 | 0.01 |
| Calcium | 0.071 | 1 |
| Chromium | 0.002 | 0.01 |
| Cobalt | 0.002 | 0.01 |
| Copper | 0.001 | 0.01 |
| Iron | 0.140 | 0.05 |
| Lead | 0.020 | 0.05 |
| Magnesium | 0.030 | 0.2 |
| Manganese | 0.001 | 0.01 |
| Molybdenum | 0.013 | 0.02 |
| Nickel | 0.005 | 0.02 |
| Potassium | 0.157 | 2 |
| Selenium | 0.078 | 0.1 |
| Silicon | 0.041 | 1 |
| Silver | 0.003 | 0.01 |
| Sodium | 0.199 | 2 |
| Strontium | 0.0009 | 0.01 |
| Thallium | 0.134 | 0.2 |
| Tin | 0.029 | 0.1 |
| Vanadium | 0.002 | 0.02 |
| Zinc | 0.004 | 0.02 |

## 3.0 SUMMARY

3.1 Before analysis can take place, samples must be digested using appropriate preparation methods. Digestion is not required when analyzing dissolved constituents as long as samples are filtered and preserved with acid.

3.2 An aerosol is created, through nebulization, and is transported to the plasma via a torch. The plasma places the elements in an atomic state which, when excited, emit a light or spectra, which is specific to that particular element. A grating separates the spectra and the intensities of the lines are measured by either photo multiplier tubes or in this case, a charged injection device. That is, the wavelength of light determines the element, and the intensity of the light omitted determines concentration.

3.3 Background light intensity must be measured adjacent to the analyte lines during analysis. The position selected for background measurement is dependent upon the complexity of the spectrum adjacent to the analytical line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not necessary when the analyte wavelength is broad and therefore may be degraded as a result of background correction.

3.4 Although, ICP-AES is typically linear over multiple orders of magnitude, samples with concentrations greater than the high calibration standard for a given element should be diluted. Samples with concentrations greater than 90% of the linear dynamic range for a given element must be diluted. Digestates containing high Silver concentration should not be diluted, but should be redigested using a lesser sample size.

3.5 Section 9.3.1 of EPA Method 200.7 revision 4.4 states, "When LRB values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained". Most of the reporting limits (PQL) for this procedure are well above 2.2 times the MDL and meet the data quality objectives of the client. As such, evaluation of the Method Blank down to 2.2 times the MDL is unnecessary as MB concentrations below the PQL are typically insignificant with respect to a detected concentration in a sample. Section 10.9 of this SOP requires that blanks are less than the PQL.

## 4.0 DEFINITIONS

4.1 Accuracy – The degree of agreement of a measured value with the true or expected value of the quantity of concern (% recovery of a known spiked analyte).

4.2 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.3 Analyte – The specific component measured in a chemical analysis.

4.4 Analytical Batch – A group of samples which are analyzed, at the instrument level, together using the same method, reagents and apparatus within the same time period. Typically, these are samples with the same batch ID in the LIMS.

4.5 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Calibration Verification Blank – An aliquot of the standard diluent (water or organic solvent) that is not carried through the sample preparation scheme. It is analyzed to verify that the analytical system is free from contamination. Also referred to as an instrument blank, ICB and CCB.

- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.

- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Preparation or Procedural Blank.

4.6 Bias – The deviation of a measured value from a known or accepted value due to matrix effects or method performance. Bias may be determined quantitatively to correct measured values. Bias may be positive or negative.

4.7 Calibration – The establishment of an analytical curve based on the absorbance, response, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type and concentration of acids, solvents, or other solutions used in the sample preparation.

4.8 Continuing Calibration Verification Standard (CCV) – A standard used to verify the continued acceptability of the initial calibration curve. A continuing calibration verification must be repeated at the beginning and end of each analytical batch and every 10-20 samples, whichever is more frequent depending on the method requirements. The concentrations of the continuing calibration verification standard shall be varied within the established calibration range. If an internal standard is used, only one continuing calibration verification must be analyzed per analytical batch.

4.9 Detection Limit – The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

- IDL – Instrument detection limit. A statistically determined detection limit used to estimate the instrument's sensitivity. The IDL is obtained by analyzing a minimum of seven consecutive blanks to assess the variability of the instrument.

- MDL – Method detection limit. The minimum concentration of a substance that can be measured and reported with a 99% degree of confidence. MDLs are determined by analyzing a minimum of seven consecutive standards that have been processed through all preparatory steps.

- PQL – The Practical Quantitation Limit is the lowest concentration that can reliably be achieved within specified limits of precision and accuracy during routine laboratory operating conditions. Typically, the PQL is a value in the range of 5 - 10 times the MDL. This is the reporting limit and is also referred to as the Estimated Quantitation Limit (EQL).

4.10 Initial Calibration Verification (ICV) – A standard used to verify the accuracy of calibration standards. Prepared from a second source than that of the calibration standards, its known value is measured against the calibration curve. This

determines the integrity of working standards. Also referred to as an external verification standard or check standard.

4.11 Interference Check Standard (ICS) – Consisting of two standard solutions (A and AB), the ICS is analyzed to verify that correct background and interelement corrections are being applied.

4.12 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.13 Laboratory Control Sample (LCS) – An aliquot of clean matrix (laboratory pure water or vendor supplied solid) spiked with target analytes or compounds representative of target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.14 Laboratory Control Sample Duplicate (LCSD) – An aliquot of clean matrix spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.15 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.16 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.17 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.18 Method of Standard Addition (MSA) – A method in which small increments of a substance under measurement are added to a sample to establish a response function, and by extrapolation, to determine the amount of the substance originally present in the sample.

4.19 Percent Recovery – A measure of accuracy that is calculated as the measured value relative to the true value, expressed as a percent.

$$\%R = \frac{MV}{TV} * 100$$

where: MV = measured value
TV = true value

4.20 Precision – The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the comparability of results from duplicate or replicate analyses. (%RPD between the recoveries of two known analyte spikes, and %RSD between the recoveries of three or more measurements).

4.21 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.22 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.23 Relative Percent Difference (% RPD) – Used to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

$$\% \text{ RPD} = \frac{|X - Y|}{(X + Y) / 2} * 100$$

where: X = value 1
Y = value 2

4.24 Relative Standard Deviation (% RSD) – Used to compare more than two values, the relative standard deviation is based on the variance and the mean of the values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

$$\% \text{ RSD} = \frac{s}{\text{avg.}} * 100$$

where: s = standard deviation
avg. = arithmetic average

4.25 Sample – A portion of material supplied by the client for analysis.

4.26 Sample Duplicate – Two aliquots of the same sample processed independently. This monitors precision of the analysis. Precision results are reported as relative percent difference (RPD).

## 5.0 INTERFERENCES

5.1 Spectral interferences are caused by overlap of spectral lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light from the line emission of high concentration elements. Spectral overlap can be compensated for by computer correction of the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line. Potential spectral interferences for recommended wavelengths have been documented but may differ for each instrument. Therefore, spectral interferences must be measured for a particular instrument by aspirating 100 mg/L of the interfering element in order to measure the false analyte concentration that can arise. A correction factor can then be calculated and stored in the computer for future corrections. For example, aspirating a 100 mg/L standard of Aluminum produces a false signal of 1.3 mg/L of Arsenic at the 193.696 line. Therefore, a correction factor can be used to compensate for this false signal.

5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Samples containing high dissolved solids can cause inaccuracies. The addition of and correction for the Yttrium internal standard (ISTD) resolves this potential interference. The ISTD option automatically corrects the instrument values for the ISTD recovery. As this correction is automatic for each sample, there are no acceptance criteria for ISTD recovery. Dilution can also reduce these inaccuracies.

5.3 Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. These effects are not significant with the ICP. They can be minimized by careful selection of operating conditions, by matrix matching and by standard addition.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Inductively coupled argon plasma emission spectrometer including a computer controlled emission spectrometer with background correction and a radio frequency generator. The software steps in sections 11.0 and 12.0 are specific to the software used on the Iris and Iris Advantage ICP-AES instruments.

7.3 Liquid Argon

7.4 Class A volumetric flasks

7.5 Class A volumetric pipettes

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

8.2.1 Lab pure water: ASTM Type II water is prepared as described in the Quality Assurance Plan.

8.2.2 Hydrochloric acid, conc. (12N HCl): Metals grade. Store this reagent in the metals preparation lab.

8.2.3 Nitric acid, conc. (18N $HNO_3$): Metals grade. Store this reagent in the metals preparation lab.

8.3 Standards

8.3.1 Stock Yttrium Internal Standard, 1000 ug/ml: Spex 8-39Y-X or equivalent. Store this standard in the metals instrument lab.

8.3.2 Working Internal Standard, 25 mg/l: In a 2L volumetric flask, dilute 50 ml of the stock internal standard to the mark with 100 ml conc. HCl, 40 ml conc. $HNO_3$, and DI water. Store this standard in the metals instrument lab.

8.3.3 Stock Calibration Standards: Store these standards in the metals instrument lab.

| VENDOR | CATALOG # | ELEMENTS | CONC., ug/ml |
|---|---|---|---|
| CPI | 4400-130317 | Sb, Mo, Si, Sn | 100 |
| CPI | 4400-130316 | K, Na | 1000 |
| | | Al, As, Ba, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Ni, Tl, Se, V, Zn | 100 |
| | | Mn, Ag, Sr | 10 |
| | | Be | 2.5 |

8.3.4 Working Calibration Standard "10": In a 100 ml volumetric flask, dilute 10.0 ml of the stock calibration standards to the mark with 2.0 ml concentrated nitric acid, 5.0 ml concentrated hydrochloric acid, and DI water. This standard is used for instrument calibration. Store this standard in the metals instrument lab.

| ELEMENTS | CONC., ug/ml |
|---|---|
| Sb, Mo, Si, Sn | 10 |
| K, Na | 100 |
| Al, As, Ba, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Ni, Tl, Se, V, Zn | 10 |
| Mn, Ag, Sr | 1.0 |
| Be | 0.250 |

8.3.5 Working Calibration Standard "5": In a 100 ml volumetric flask, dilute 5.0 ml of the stock calibration standards to the mark with 2.0 ml concentrated nitric acid, 5.0 ml concentrated hydrochloric acid, and DI water. This standard is used for instrument calibration. Store this standard in the metals instrument lab.

| ELEMENTS | CONC., ug/ml |
|---|---|
| Sb, Mo, Si, Sn | 5 |
| K, Na | 50 |
| Al, As, Ba, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Ni, Tl, Se, V, Zn | 5 |
| Mn, Ag, Sr | 0.500 |
| Be | 0.125 |

8.3.6 Working Calibration Standard "2": In a 100 ml volumetric flask, dilute 2.0 ml of the stock calibration standard CPI 4400-130316 to the mark with 2.0 ml concentrated nitric acid, 5.0 ml concentrated hydrochloric acid, and DI water. This standard is used for calibrating the 249.3nm wavelength Iron line. Store this standard in the metals instrument lab.

| ELEMENTS | CONC., ug/ml |
|---|---|
| Fe | 2 |

8.3.7 ICP Standard "1": Prepared identical to "Working Calibration Standard 10", this solution is used to verify the calibration at the highest standard concentration. Store this standard in the metals instrument lab.

8.3.8 Stock Verification Standards: Store this standard in the metals instrument lab.

| VENDOR | CATALOG # | ELEMENTS | CONC., ug/ml |
|---|---|---|---|
| Inorganic Ventures | QCP-CICV-1 | Ca, Mg, K, Na | 2500 |
| | | Al, Ba | 1000 |
| | | Fe | 500 |
| | | Co, Mn, Ni, V, Zn | 250 |
| | | Cu, Ag | 125 |
| | | Cr | 100 |
| | | Be | 25 |
| Inorganic Ventures | QCP-CICV-2 | Sb | 500 |
| Inorganic Ventures | QCP-CICV-3 | As, Pb, Se, Tl | 500 |
| | | Cd | 250 |

8.3.9 Stock Single Element Verification Standards: Obtain from an approved vendor. Na at 1000 ug/ml, B, Mo, Si and Sn at 10000 ug/ml, and Sr at 1000 ug/ml.

8.3.10 Intermediate ICV/CCV Solution: Prepare the following dilutions using the various single-element stock standards diluted to 100 ml. Store this standard in the metals instrument lab.

| ELEMENTS | STOCK CONC., ug/ml | VOL. STOCK, ml | FINAL CONC., ug/ml |
|---|---|---|---|
| B, Mo, Si, Sn | 10,000 | 5 | 500 |
| Sr | 1000 | 10 | 100 |

8.3.11 Working ICV Standard: In a 100 ml volumetric flask, combine and dilute 0.2 ml of the stock verification standards (8.3.8), 0.2 ml of the ICV/CCV solution (8.3.10), and 0.5 ml of the 1000 ppm Na standard (8.3.9) to the mark with 5 ml conc. HCl, 2 ml conc. $HNO_3$, and DI water. Store this standard in the metals instrument lab. This produces a second source verification standard of:

| ELEMENTS | CONC., ug/ml |
|---|---|
| Na | 10 |
| Ca, Mg, K | 5.0 |
| Al, Ba | 2.0 |
| Fe, Sb, As, Pb, Se, Tl, B, Mo, Si, Sn | 1.0 |
| Co, Mn, V, Zn, Cd | 0.5 |
| Cu, Ag | 0.25 |
| Cr, Sr | 0.20 |
| Be | 0.05 |

8.3.12 Working CCV Standard: In a 500 ml volumetric flask, combine and dilute 5.0 ml of the stock verification standards (8.3.8) and the ICV/CCV solution (8.3.10) to the mark with 25 ml conc. HCl, 10 ml conc. $HNO_3$, and DI water. Store this standard in the metals instrument lab. This produces a second source verification standard of:

| ELEMENTS | CONC., ug/ml |
|---|---|
| Ca, Mg, K, Na | 25 |
| Al, Ba | 10 |
| Fe, Sb, As, Pb, Se, Tl, B, Mo, Si, Sn | 5.0 |
| Co, Mn, V, Zn, Cd | 2.5 |
| Cu, Ag | 1.25 |
| Cr, Sr | 1.0 |
| Be | 0.25 |

8.3.13 Stock Interferents A Standard: Contains Al, Ca, and Mg at 5000 ug/ml, and Fe at 2000 ug/ml. Store this standard in the metals instrument lab.

8.3.14 Stock Analytes B Standard: Spex #INT-B1 or equivalent contains Ag, Cd, Ni, Pb, and Zn at 100 ug/ml and Ba, Be, Co, Cr, Cu, Mn, and V at 50 ug/ml. Store this standard in the metals instrument lab.

8.3.15 Stock Single-element Standards: Obtain from an approved vendor. Store these standards in the metals instrument lab.

| ELEMENTS | CONC., ug/ml |
|---|---|
| K, Na | 10,000 |
| As, B, Mo, Sr, Tl, Sb, Se | 1000 |

8.3.16 Intermediate ICSAB Solution 1: In a 100 ml volumetric flask, dilute the various single-element stock standards (8.3.15) to the mark with 1 ml conc. $HNO_3$ and DI water. Store this standard in the metals instrument lab.

| ELEMENTS | STOCK CONC., ug/ml | VOL. STOCK, ml | FINAL CONC., ug/ml |
|---|---|---|---|
| K, Na | 10,000 | 10 | 1000 |
| As, B, Mo, Sr, Tl, Sb, Se | 1000 | 10 | 100 |

8.3.17 Stock Single Element Tin Standard, 1000 ug/ml: Obtain from an approved vendor. Store this standard in the metals instrument lab.

8.3.18 Intermediate ICSAB Solution 2, 100 ug/ml: In a 100 ml volumetric flask, dilute 10 ml of the stock Sn standard (8.3.17) to the mark with 5 ml conc. $HNO_3$ and DI water. Store this standard in the metals instrument lab.

8.3.19 Working Interference Check Standard (ICSA): In a 500 ml volumetric flask, dilute 50 ml of the stock Interferents A standard to the mark with 25 ml conc. HCl, 10 ml conc. $HNO_3$, and DI water. This prepares an interference check standard of 500 ug/ml Al, Ca, and Mg, and 200 ug/ml Fe. Store this standard in the metals instrument lab.

8.3.20 Working Interference Check Standard (ICSAB+): In separate 500 ml volumetric flasks, prepare the following dilutions diluted to volume with 25 ml conc. HCl, 10 ml conc. $HNO_3$, and DI water. Store this standard in the metals instrument lab.

| STANDARD | VOL. STANDARD, ml | FINAL CONC., ug/ml |
|---|---|---|
| Stock Interferents A | 50 | Al, Ca, Mg @ 500; Fe @ 200 |
| Stock Analytes B | 5 | Ag, Cd, Ni, Pb, Zn @ 1; Ba, Be, Co, Cr, Cu, Mn, V @ 0.5 |
| Intermediate ICSAB Solution 1 | 5 | K, Na @ 10; Sb, As, B, Mo, Se, Sr, Tl @ 1 |
| Intermediate ICSAB Solution 2 | 5 | Sn @ 5 |
| Stock Single Element Standards (K and Na at 10000, Tl at 1000) | 2 | K, Na @ 40; Tl @ 4 |

*NOTE: The final concentration of K and Na is 50 ug/ml, and 5 ug/ml for Tl.

8.3.21 LCS/MS: When prepared as detailed in the preparation SOPs, the final concentrations are listed below. For solids samples, the vendor supplies the final concentration of the LCS. The spiked concentrations are listed in the Specs tab of the appropriate test code.

| Aqueous LCS/MS | Conc., mg/l | Aqueous LCS/MS | Conc., mg/l |
|---|---|---|---|
| Aluminum | 2 | Manganese | 0.5 |
| Antimony | 0.5 | Molybdenum | 0.4 |
| Arsenic | 2 | Nickel | 0.5 |
| Barium | 2 | Potassium | 20 |
| Beryllium | 0.05 | Selenium | 2 |
| Boron | 0.4 | Silicon | 0.4 |
| Cadmium | 0.05 | Silver | 0.05 |
| Calcium | 20 | Sodium | 20 |
| Chromium | 0.2 | Strontium | 0.4 |
| Cobalt | 0.5 | Thallium | 2 |
| Copper | 0.25 | Tin | 0.5 |
| Iron | 1 | Titanium | 0 |
| Lead | 0.5 | Vanadium | 0.5 |
| Magnesium | 20 | Zinc | 0.5 |

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Water samples should be collected in a plastic container. Chemical preservation consists of $HNO_3$ to pH <2 for water samples only. Samples are stored on the shelves in the metals preparation lab. Prior to sample preparation, samples must be held for a minimum of 16 hours after preservation. Solid samples should be retained at 4°C until prepared and are stored in the main Walk-in Cooler. Digestates are stored in plastic sample cups with lids or capped digestion tubes, and are placed in the metals instrument lab for analysis.

9.3 Analysis must be performed within the maximum allowable hold time of 6 months from collection.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Analyze four separate standards prepared in the range of 8-10 times the method detection limit listed in section 2.1. These standards must be from a source different from that used for calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.2 An *Instrument Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Analyze a minimum of seven (maximum of ten) calibration blanks. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.3 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Analyze a minimum of seven (maximum of ten) standards prepared in the range of 2-5 times the method detection limit listed in section 2.1 or an estimated detection limit. These standards must be taken through the entire analytical procedure. Submit the data to the QA department for evaluation.

10.4 A *Linear Dynamic Range* study must be performed on an annual basis. Analyze a series of standards in increasing concentration (i.e. 50 mg/l, 100 mg/l, 150 mg/l, etc.). The LDR is defined as the concentration at which the recovery varies by more than 10% from the true value. Refer to the Capability and Detection Limit Studies SOP for details.

10.5 Analysis of the *High Calibration Standard (ICP Standard 1)* must be analyzed immediately after calibration. Acceptance criteria are 90.0 – 110% recovery. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate.

10.6 An *Initial Calibration Verification (ICV) Standard* must be analyzed immediately after verification with the high calibration standard. Acceptance criteria are 95.0 – 105% recovery for Method 200.7 and 90.0 – 110% recovery for Method 6010B. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the

acceptance criteria, stop analysis and recalibrate. Samples associated with a verification that fails with positive bias can be reported if the sample concentration is a non-detect. The LIMS will flag all failed ICV recoveries with a "S" qualifier.

10.7 An *Initial Calibration Blank (ICB)* sample must be analyzed after the ICV. The acceptance criteria are the absolute value of the blank less than the absolute value of the PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. If the blank does not meet the acceptance criteria, all samples < PQL or greater than 10 times the blank contamination may be reported. All other environmental samples must be reanalyzed. If insufficient sample or hold time is available for reanalysis, report the data with a "B" qualifier as defined in the LIMS.

10.8 The *Interference Check Standards* (solutions A and AB+) must be analyzed at the beginning and end of each analytical batch. Acceptance criteria for the ICSA are 80.0 – 120% recovery for Al, Ca, Mn and Fe, and <PQL for the other elements. Acceptance criteria for the ICSAB+ are 80.0 – 120% recovery for all spiked elements. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier. Samples associated with a verification that fails with positive bias can be reported if the sample concentration is a non-detect or if there are no interfering elements measured in the sample. The LIMS will flag all failed ICS recoveries with a "S" qualifier.

10.9 A *Method Blank* must be analyzed with each batch of maximum 20 samples and at a minimum of one per 24-hour calendar day. Acceptance criteria are < PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis, recalibrate and reanalyze. If the blank does not meet the acceptance criteria, all samples < PQL or greater than 10 times the blank contamination may be reported. All other environmental samples must be redigested and analyzed. If insufficient sample or hold time is available for reanalysis, report the data with a "B" qualifier as defined in the LIMS. NOTE: See section of this SOP for additional information regarding the MB criteria.

10.10 A *Laboratory Control Sample* must be analyzed with each batch of maximum 20 samples and at a minimum of one per 24-hour calendar day. Acceptance criteria are 85.0 – 115% recovery for waters and the vendor supplied recovery limits for solids (which are identified within the test code in the LIMS). Only the elements of interest must meet this criteria. If the acceptance criteria, stop analysis, recalibrate and reanalyze. If the LCS fails with high bias, samples having a non-detectable concentration may be reported. If reanalysis fails to meet the acceptance criteria all environmental samples must be reprepared and analyzed. If insufficient sample or hold time is available for reanalysis, report the data with a "S" qualifier as defined in the LIMS.

10.11 A *Continuing Calibration Verification (CCV) Standard* must be analyzed immediately after every ten samples and after the last sample. Acceptance criteria are 90.0 – 110% recovery. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. Samples associated with a verification that fails with positive bias can be reported

if the sample concentration is a non-detect. The LIMS will flag all failed CCV recoveries with a "S" qualifier.

10.12 A *Continuing Calibration Blank (CCB)* sample must be analyzed with every CCV. The acceptance criteria are the absolute value of the blank less than the absolute value of the PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. If the blank does not meet the acceptance criteria, all samples < PQL or greater than 10 times the blank contamination may be reported. All other environmental samples must be reanalyzed. If insufficient sample or hold time is available for reanalysis, report the data with a "B" qualifier as defined in the LIMS.

10.13 A *Matrix Spike and Matrix Spike Duplicate* sample must be analyzed with each batch of maximum 10 samples and at a minimum of one per day. Acceptance criteria for accuracy are 70.0 – 130 %R. Acceptance criteria for precision are ≤ 20.0% RPD (waters) and ≤ 30.0% RPD (solids). Either the MS or MSD must meet the accuracy criteria. If the accuracy or precision criteria are not met, reanalyze. If reanalysis fails to meet the accuracy criteria only, perform and evaluate a PDS on that sample. If not acceptable, the sample and its MS/MSD must be reprepared and analyzed. If the precision criteria are not met, all samples in that batch must be reprepared and analyzed. If insufficient sample or hold time is available for reanalysis, report the data with a "S" qualifier (for accuracy failure) or a "R" qualifier (for precision failure) as appropriate. If insufficient sample is available for the preparation of a MS/MSD, a MS on two separate samples or a LCSD may be performed to provide for precision assessment.

10.14 A *Post Digestion Spike* can be analyzed on any sample to evaluate the potential of matrix interference. Acceptance criteria are 85.0 – 115% recovery for Method 200.7 and 75.0 – 125% recovery for Method 6010B. Use the recovery data to help evaluate any bias as detailed in the MS/MSD Corrective Action Flowchart. The LIMS will flag all failed PDS recoveries with a "S" qualifier.

10.15 A *Serial Dilution* can be analyzed on any sample to evaluate the potential of matrix interference. Acceptance criteria for a 1:5 dilution are ≤ 10% difference between the original and the diluted sample concentration. If acceptance criteria are not met, another serial dilution of a different factor (e.g. 1:10) may be analyzed. The LIMS will flag all failed SD recoveries with a "R" qualifier.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Perform the required preventative maintenance as necessary. This includes a daily check of the autosampler components, a monthly changing of the tubing, and a semi-annual cleaning of the filters. Gas flow checks, and nebulizer cleanings are conducted as needed. All preventative maintenance is documented in the appropriate PM logbook.

11.2 A new calibration must be performed for each analytical batch. The calibration, QC samples and environmental samples are linked together through the

autosampler/run table number, which is printed on each page of the instrument printout. Instrument printouts are retained in chronological order.

11.3 Follow the steps in section 12.0, below. Unless you are running an unattended autosampler batch of samples, verify that the initial quality control (ICV, ICB, ICS) is acceptable before continuing with sample analysis. Analysis of environmental samples cannot proceed without the generation of an acceptable calibration. A typical sequence follows the order of Calibration, High Cal Standard (ICP Standard 1), ICV, ICB, ICSA, ICSAB+, up to 10 batch/matrix QC samples and environmental samples, CCV, CCB, etc.

## 12.0 PROCEDURE

### 12.1 Typical Calibration and Analysis

12.1.1 Make certain that the hoods are turned on and the waste buckets are empty.

12.1.2 Turn on the computer and the printer.

12.1.3 Turn on the instrument and the autosampler.

12.1.4 Open the Thermospec software and click the "flame" icon.

12.1.5 Click the Reset Controller button to perform a hard reset of the camera and the instrument.

12.1.6 Verify that the pump tubing cassettes are locked and that there is rinse water in the rinse station.

12.1.7 Click the Ignite button and then click OK to ignite the torch.

12.1.8 After the torch is lit, check the flow of the rinse water, internal standard and sample lines. Adjust as necessary to obtain a smooth flow. (The pump is not a variable speed pump, so the flow needs to only be smooth).

12.1.9 Allow the instrument a minimum of 30 minutes to warm up before calibration.

12.1.10 Click Method (at the top of the screen) and open the appropriate method.

12.1.11 Set up the autosampler tray.

12.1.11.1 To set up the autosampler tray, click Run then choose the Automated Analysis option.

12.1.11.2 Click Samples then the Test Tube + icon.

12.1.11.3 Enter the sample number in the Sample Name field. In the field labeled Lab ID, enter in the sample type (e.g. MBLK, LCS, SAMP, MS). In the Cust Samp ID field, enter the test code for that sample. The "200.7" series of test codes should be used for the instrument and batch QC samples as the 200.7 series has more stringent acceptance criteria. NOTE: the information entered in these fields must be exactly as in the LIMS otherwise the automated data transfer will not work properly.

12.1.11.4 Click Done when finished entering the information for this sample and continue entering the information for the remainder of the run.

12.1.11.5 When all samples have been added to the autosampler tray, click Save.

12.1.11.6 Click Samples (at the top of the screen) and save the autosampler tray named in a format that identifies the instrument and date of that tray. (For the Iris, ICP #1, name the file Immdd-*, where the "I" signifies ICP #1, mm is the month, dd is the date, and * is a number or letter indicating the sequential number of the run for that day. That is –1 or –A indicates the first tray of the day, -2 or –B indicates the second run of that day, etc. For the Iris Advantage, ICP #2, name the file in the format of Ammo-*).

12.1.12 Close the Samples box.

12.1.13 Click the New button on the Automated Analysis screen.

12.1.14 Click the Autosampler + icon and, using the drop down menus, set the Protocol to Complete.

12.1.15 Choose the appropriate method (step 12.10) and autosampler tray number (step 12.11.6) for the autosampler run. Change the rinse time to 55 seconds then click OK.

12.1.16 Click Table (at the top of the screen) and save it with the same name as the autosampler tray (12.11.6).

12.1.17 Click Run (at the top of the screen), Setup, and then the diamond shaped icon next the "Samples". This will show the autosampler locations of the standards and samples. Print this list.

12.1.18 Load the standards and samples into the appropriate locations on the autosampler.

12.1.19 Click OK then Run to begin the analysis.

**12.2 Reprofiling the Instrument**

12.2.1 The instrument profile must be re-established if it is lost during sample analysis. Profile the instrument by performing the following steps.

12.2.2 Aspirate the working calibration standard "10".

12.2.3 Click "Instrument" at the top of the screen then choose Auto Peak Adjust followed by Run. The instrument will search for the wavelengths listed in the method and verify their mapping.

12.2.4 When this search is finished, any wavelengths not found will be shown on the screen. Write these elements and wavelengths on a piece of paper.

12.2.5 Map any missing wavelengths by clicking "Instrument" at the top of the screen then choosing Research.

12.2.6 On the Research screen, choose Image followed by New and aspirate the working calibration standard "10".

12.2.7 Change the sample flush time to 40 seconds and choose Run.

12.2.8 After the standard has been analyzed, a map will appear on the screen. Choose "Add Maps" on the toolbar at the top of the screen.

12.2.9 Select the element to correct. Squares will appear on the map you created showing the wavelengths for the element you have chosen. Find the particular wavelength you are looking for and observe where it is in relation to the peak that was found. Move the square position to that peak by pressing the Ctrl key and doing a left-click on the mouse. Repeat this step for all other missing wavelengths.

12.2.10 When all wavelengths have been correctly mapped, perform another Auto Peak Adjust. This Auto Peak Adjust and mapping process must be repeated until all wavelengths are automatically found.

12.2.11 Calibrate the instrument calibration as described above.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 After review, enter final results into the LIMS system. Results flagged by the LIMS with an "E" qualifier are above the linear range of the instrument. There is less certainty in these data and, if sufficient sample and holding time are available, should be reanalyzed at an appropriate dilution. Details on the procedure for entering analytical data are in the Data Entry SOP.

13.2 The instrument calculates the sample concentration using linear regression based on the calibration curve and the following calculation:

Concentration, ppm = (instrument reading, mg/l) ($V_f$) (DF) / SS

where: $V_f$ = final digested volume, ml
DF = dilution factor
SS = sample size digested, ml or g

13.3 The LIMS calculates the dry-weight concentration for solid samples as follows:

$$\text{Conc. Dry} = \frac{\text{(wet weight conc.)}}{\text{(100 - \%Moisture)}}$$

## 14.0 METHOD PERFORMANCE

14.1 See section 18.0 for Initial Demonstration of Capability data. Method Detection Limits are listed in section 2.1.

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Refer to the SIMALABS International Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

## 17.0 REFERENCES

17.1 USEPA Method 200.7, revision 4.4

17.2 SW-846 Method 6010B

17.3 SIMALABS International Quality Assurance Plan, current revision

17.4 SIMALABS International SOP Preparation of Aqueous Samples and Extracts for Total Metals Analysis by Inductively Coupled Plasma or Flame Atomic Absorption Spectroscopy, current revision

17.5 SIMALABS International SOP Preparation of Non-Aqueous Samples and Extracts for Total Metals Analysis, current revision

17.6 SIMALABS International SOP Toxicity Characteristic Leaching Procedure for Metals and Semi-Volatile Organic Compounds using SW-846 Method 1311, current revision

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

A typical IDC study will yield data similar to:

| Analyte | Spiked Value | Average %R | %R Criteria | %RSD | RSD Criteria |
|---|---|---|---|---|---|
| Aluminum | 2 | 95.6 | 80-120 | 1.34 | 20 |
| Antimony | 0.5 | 105 | 80-120 | 1.49 | 20 |
| Arsenic | 2 | 99.3 | 80-120 | 1.02 | 20 |
| Barium | 2 | 102 | 80-120 | 0.55 | 20 |
| Beryllium | 0.05 | 91.1 | 80-120 | 0.85 | 20 |
| Boron | 0.4 | 97.5 | 80-120 | 1.54 | 20 |
| Cadmium | 0.05 | 93.4 | 80-120 | 1.08 | 20 |
| Calcium | 20 | 98.3 | 80-120 | 0.73 | 20 |
| Chromium | 0.2 | 101 | 80-120 | 0.30 | 20 |
| Cobalt | 0.5 | 103 | 80-120 | 0.96 | 20 |
| Copper | 0.25 | 100 | 80-120 | 0.80 | 20 |
| Iron | 1 | 105 | 80-120 | 2.31 | 20 |
| Lead | 0.5 | 103 | 80-120 | 1.75 | 20 |
| Magnesium | 20 | 104 | 80-120 | 0.89 | 20 |
| Manganese | 0.5 | 100 | 80-120 | 0.65 | 20 |
| Molybdenum | 0.4 | 104 | 80-120 | 2.24 | 20 |
| Nickel | 0.5 | 100 | 80-120 | 0.27 | 20 |
| Potassium | 20 | 95.5 | 80-120 | 1.10 | 20 |
| Selenium | 2 | 109 | 80-120 | 0.55 | 20 |
| Silicon | 0.4 | 110 | 80-120 | 2.07 | 20 |
| Silver | 0.05 | 82.1 | 80-120 | 1.99 | 20 |
| Sodium | 20 | 98.3 | 80-120 | 1.45 | 20 |
| Strontium | 0.4 | 99.2 | 80-120 | 0.80 | 20 |
| Thallium | 2 | 155 | 80-120 | 1.43 | 20 |
| Tin | 0.5 | 106 | 80-120 | 2.90 | 20 |
| Vanadium | 0.5 | 99.5 | 80-120 | 0.85 | 20 |
| Zinc | 0.5 | 104 | 80-120 | 0.70 | 20 |

# STANDARD OPERATING PROCEDURE FOR MERCURY USING THE CETAC M6000A BY EPA METHOD 245.1 AND SW-846 METHODS 7470A/7471A

Originating Author: Jeff Loewe
Revision Author: Troy Goehl

This SOP is effective upon signed approval by the following:

Troy Goehl — Unit Supervisor — 4-8-2002 — Date

Jeff M. Loewe — QA/QC Director — 4-4-2002 — Date

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

# 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This is a cold vapor procedure for the determination of mercury. This procedure is applicable to the analysis of aqueous, non-aqueous liquid, drinking water, and solid matrix samples. The routine reporting limits (PQL) are 0.2 ug/l and 10 ug/g.

## 3.0 SUMMARY

3.1 The mercury is reduced to the elemental state through digestion with acids and oxidizers. The digestate is aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance of the mercury vapor at 253.7-nm is measured as a function of mercury concentration.

3.2 The linear working range is 0.2 – 10 ug/l.

3.3 The preparation of the stannous chloride solution deviates from the reference method. EPA method 245.1 requires the use of sulfuric acid ($H_2SO_4$) which results in a turbid solution. This turbidity adversely effects the performance of the analyzer. The use of hydrochloric acid (HCl), however, produces a clear solution, increases the stability of the solution, and produces results of equal quality, as seen in the IDL, MDL, and QC sample analyses.

## 4.0 DEFINITIONS

4.1 Accuracy – The degree of agreement of a measured value with the true or expected value of the quantity of concern (% recovery of a known spiked analyte).

4.2 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.3 Analyte – The specific component measured in a chemical analysis.

4.4 Analytical Batch – A group of samples which are analyzed, at the instrument level, together using the same method, reagents and apparatus within the same time period. Typically, these are samples in the same run ID in the LIMS.

4.5 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Calibration Blank – An aliquot of the standard diluent (water or organic solvent) that is not carried through the sample preparation scheme. It is analyzed to verify that the analytical system is free from contamination. Also referred to as an instrument blank or solvent blank.

- Method Blank – An aliquot of lab pure water taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Procedural Blank.

4.6 Bias – The deviation of a measured value from a known or accepted value due to matrix effects or method performance. Bias may be determined quantitatively to correct measured values. Bias may be positive or negative.

4.7 Calibration – The establishment of an analytical curve based on the absorbance, response, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type and concentration of acids, solvents, or other solutions used in the sample preparation.

4.8 Continuing Calibration Verification Standard (CCV) – A standard used to verify the continued acceptability of the initial calibration curve. A continuing calibration verification must be analyzed after every 10 and the last environmental sample. The concentration of the continuing calibration verification standard shall be varied from the concentration of the initial calibration verification standard

4.9 Detection Limit – The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

- IDL – Instrument detection limit. A statistically determined detection limit used to estimate the instrument's sensitivity. The IDL is obtained by analyzing seven consecutive blanks to assess the variability of the instrument.

- MDL – Method detection limit. The minimum concentration of a substance that can be measured and reported with a 99% degree of confidence. MDLs are determined by analyzing a minimum of seven consecutive standards that have been processed through all preparatory steps.

- PQL – The Practical Quantitation Limit is the lowest concentration that can reliably be achieved within specified limits of precision and accuracy during routine laboratory operating conditions. Typically, the PQL is a value in the range of 5 - 10 times the MDL. This is the reporting limit and is also referred to as the Estimated Quantitation Limit (EQL).

4.10 Initial Calibration Verification (ICV) – A standard used to verify the accuracy of calibration standards. Prepared from a second source than that of the calibration standards, its known value is measured against the calibration curve. This determines the integrity of working standards. Also referred to as an external verification standard or check standard.

4.11 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.12 Laboratory Control Sample (LCS) – An aliquot of clean matrix (lab pure water or vendor supplied solid) spiked with target analyte. The sample is carried through the

entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.13 Laboratory Control Sample Duplicate (LCSD) – An aliquot of clean matrix (lab pure water or vendor supplied solid) spiked with the identical amount of target analyte as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.14 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.15 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.16 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.17 Percent Recovery – A measure of accuracy that is calculated as the measured value relative to the true value, expressed as a percent.

$$\%R = \frac{MV}{TV} * 100$$

where: MV = measured value
TV = true value

4.18 Precision – The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the comparability of results from duplicate or replicate analyses. (%RPD between the recoveries of two known analyte spikes, and %RSD between the recoveries of three or more measurements).

4.19 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.20 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.21 Relative Percent Difference (% RPD) – Used to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

$$\% \text{ RPD} = \frac{|X - Y|}{(X + Y) / 2} * 100$$

where: X = value 1
Y = value 2

4.22 Sample – A portion of material to be analyzed.

- Environmental sample – sample supplied by the client for analysis.
- QC sample – sample prepared in the lab analyzed to assess the bias/precision of the analytical system.

## 5.0 INTERFERENCES

5.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/l of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

5.2 Copper has also been reported to interfere, however, copper concentrations as high as 10 mg/l had no effect on recovery of mercury from spiked samples.

5.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 ml) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 ml).

5.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Digestion block capable of maintaining 95°C (digestion block may not be used for the digestion of samples applicable to North Carolina certification)

7.3 Water bath capable of maintaining 95°C (used only for samples applicable to North Carolina certification)

7.4 Plastic digestion tubes, Environmental Express Catalog #SC475

7.5 CETAC M6000A Mercury analyzer

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

All reagents are stored in the metals preparation lab unless otherwise noted.

8.2.1 Lab pure water: Analyte free water is prepared as described in the Quality Assurance Plan.

8.2.2 Hydrochloric acid, concentrated HCl

8.2.3 Nitric acid, concentrated $HNO_3$

8.2.4 Sulfuric acid, concentrated $H_2SO_4$

8.2.5 Aqua regia: Mix 3 parts of conc. HCl and 1 part conc. $HNO_3$. CAUTION: This solution is very corrosive. Handle with care. Prepare this reagent immediately before use.

8.2.6 Potassium persulfate, $K_2S_2O_8$: Fisher catalog #P282-500 or equivalent.

8.2.7 Potassium persulfate reagent: In a 1L volumetric flask, dissolve and dilute 50g $K_2S_2O_8$ to the mark with DI water.

8.2.8 Potassium permanganate, $KMnO_4$: Fisher catalog #P279-212 or equivalent.

8.2.9 Potassium permanganate reagent: In a 1L volumetric flask, dissolve and dilute 50g $KMnO_4$ to the mark with DI water.

8.2.10 Sodium chloride, NaCl: Fisher catalog #S271-3 or equivalent.

8.2.11 Hydroxylamine hydrochloride, $NH_2OH{\cdot}HCl$: Fisher catalog #H330-1 or equivalent.

8.2.12 Sodium chloride - hydroxylamine hydrochloride reagent: In a 500-ml volumetric flask, dissolve and dilute 60g NaCl and 60g $NH_2OH{\cdot}HCl$ to the mark with DI water.

8.2.13 Stannous chloride, $SnCl{\cdot}2H_2O$: Fisher catalog #T142-500 or equivalent.

8.2.14 Stannous chloride reagent: In a 1L volumetric flask, dissolve 100g $SnCl{\cdot}2H_2O$ in 70-ml conc. HCl. When completely dissolved, dilute to the mark with DI water.

8.3 Standards

All reagents are stored in the metals preparation lab unless otherwise noted.

8.3.1 Stock Calibration Standard, 1000 mg/l: Spex #PLHG4-24 or equivalent

8.3.2 Intermediate Calibration Standard, 10 mg/l: In a 100-ml volumetric flask, dilute 1.0-ml of the stock calibration standard to the mark with 2-ml conc. $HNO_3$ and DI water.

8.3.3 Working Calibration Standard, 20 ug/l: In a 500-ml volumetric flask, dilute 1.0-ml of the intermediate calibration standard to the mark with 2-ml conc. $HNO_3$ and DI water.

8.3.4 Calibration curve: As separate samples, prepare the calibration standards by processing the following volumes of the working calibration standard.

| Vol. Working Cal. Std., ml | Final Conc., ug/l |
|---|---|
| 0 | 0 |
| 0.25 | 0.2 |
| 1.25 | 1.0 |
| 2.50 | 2.0 |
| 6.25 | 5.0 |
| 12.5 | 10.0 |

8.3.5 Stock Verification Standard, 20 mg/l: Inorganic Ventures #TCLP-AA-Hg or equivalent.

8.3.6 Intermediate Verification Standard, 1 mg/l: In a 100-ml volumetric flask, dilute 5.0-ml of the stock verification standard to the mark with 2-ml conc. $HNO_3$ and DI water.

8.3.7 Working Verification Standard, 10 ug/l: In a 500-ml volumetric flask, dilute 5-ml of the intermediate verification standard to the mark with 2-ml conc. $HNO_3$ and DI water.

8.3.8 ICV, 1 ug/l: In a 25-ml volumetric flask, dilute 2.5-ml of the working verification standard to the mark with DI water and process this standard as a sample.

8.3.9 CCV, 5 ug/l: In a 25-ml volumetric flask, dilute 12.5-ml of the working verification standard to the mark with DI water and process this standard as a sample.

8.3.10 LCS/MS Spiking Solution, 100 ug/l: In a 10-ml volumetric flask, dilute 1.0-ml of the intermediate verification standard to the mark with 0.5-ml conc. $HNO_3$ and DI water.

8.3.11 LCS/MS, 2 ug/l: Add 0.5-ml of the LCS/MS spiking solution to 25-ml DI water or sample to prepare the LCS or MS, respectively, and process as a sample.

8.3.12 Solid LCS: ERA catalog #540

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Water samples should be collected in a plastic container. Preservation consists of $HNO_3$ to pH $\leq$ 2. Samples are stored on the sample storage shelves in the metals preparation lab. Solid samples should be collected in a glass or plastic container. Preservation consists of storage in the range of 0.1-6°C. Samples are stored in the main sample storage cooler.

9.3 Analysis must be performed within the maximum allowable hold time of 28 days from collection.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Analyze four separate standards prepared in the range of 8-10 times the method detection limit listed in section 14.0. These standards must be from a source different from that used for calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.2 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Analyze a minimum of seven (maximum of ten) standards prepared in the range of 2-5 times the method detection limit listed in section 14.0 or an estimated detection limit. These standards must be taken through the entire analytical procedure. Submit

the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.3 *PQL Verification* must be performed when analyzing samples applicable to our North Carolina certification. This is accomplished by analyzing the 0.2 ug/l calibration standard after the calibration curve is established. Acceptance criteria are the nominal limits of 75.0 – 125% recovery, as set forth by the NC DENR. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. Samples associated with a verification that fails with positive bias can be reported if the sample concentration is a non-detect. If insufficient holding time or sample size remains for reanalysis, results can be reported with a Case Narrative noting the failure of the control sample.

10.3.1 NOTE: An acceptable alternative to physically analyzing the PQL standard is to calculate the theoretical concentration based on the average response of that standard in the calibration curve data. To do this, enter the calibration data into a calculator (or Excel spreadsheet) capable of linear regression statistical calculations then enter the uabs of the 0.2 ug/l standard from the calibration curve. This data is used to forecast/predict the concentration with respect to the curve. The acceptance criteria for this technique are the same as if the standard were physically analyzed as a sample. Document the recovery on the instrument printout.

10.4 An *Initial Calibration Verification (ICV) Standard* must be analyzed immediately after calibration. Acceptance criteria are 90.0 – 110% recovery. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. Samples associated with a verification that fails with positive bias can be reported if the sample concentration is a non-detect. If insufficient holding time or sample size remains for reanalysis, results can be reported with a Case Narrative noting the failure of the control sample.

10.5 A *Continuing Calibration Verification (CCV) Standard* must be analyzed after every 10 environmental samples and after the last environmental sample. Acceptance criteria are 80.0 – 120% recovery. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. Samples associated with a verification that fails with positive bias can be reported if the sample concentration is a non-detect. If insufficient holding time or sample size remains for reanalysis, results can be reported with a Case Narrative noting the failure of the control sample.

10.6 A *Calibration Verification Blank (ICB/CCB)* sample must be analyzed after each calibration verification standard. The acceptance criteria are the absolute value of the blank less than the absolute value of the PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate. If the blank does not meet the acceptance criteria, all samples < PQL or greater than 10 times the blank contamination may be reported with a "B" qualifier as defined in the LIMS. All other environmental samples must be reanalyzed. If insufficient holding time or sample size remains for reanalysis,

results can be reported with a Case Narrative noting the failure of the control sample.

10.7 A *Method Blank* must be analyzed with each batch of maximum 20 samples and at a minimum of one per 24-hour calendar day. Acceptance criteria are < PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, all samples < PQL or greater than 10 times the blank contamination may be reported with a "B" qualifier as defined in the LIMS. All other environmental samples must be reanalyzed. If insufficient holding time or sample size remains for reanalysis, results can be reported with a Case Narrative noting the failure of the control sample.

10.8 A *Laboratory Control Sample* must be analyzed with each batch of maximum 20 samples and at a minimum of one per 24-hour calendar day. Acceptance criteria are the statistical recovery limits of 85.0 - 115% for waters and the vendor supplied recovery limits for solids. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria all environmental samples must be reprepared and analyzed. If the LCS fails with high bias, samples having a non-detectable concentration may be reported without qualification. If insufficient holding time or sample size remains for reanalysis, results can be reported with a Case Narrative noting the failure of the control sample.

10.9 A *Matrix Spike and Matrix Spike Duplicate* sample must be analyzed with each batch of maximum 10 samples and at a minimum of one per 24-hour calendar day. Acceptance criteria are 70.0 – 130% recovery for accuracy, and ≤ 20.0% RPD for precision. Either the MS or MSD must meet the accuracy criteria. If the accuracy criteria are not met, evaluate the precision criteria. If the precision criteria are met, reprepare and analyze the sample or prepare and analyze a PDS on that sample (the choice is left to analyst discretion). If reanalysis fails to meet the accuracy criteria and a PDS was not performed, perform and evaluate a PDS on that sample. If the PDS recovery does not meet the acceptance criteria and the precision criteria are met, complete a Corrective Action Report (CAR) Form. This data may be reported with a Case Narrative notifying the client of the control failure. If the precision criteria are not met, all samples in that preparation batch must be reprepared and analyzed. If insufficient sample or hold time is available for reanalysis, report the data with a "S" qualifier (for accuracy failure) or a "R" qualifier (for precision failure) as appropriate. If insufficient sample is available for the preparation of a MS/MSD, a MS on two separate samples or a LCSD must be performed to provide for precision assessment.

10.10 A *Post Digestion Spike (PDS)* can be analyzed on any sample to evaluate the potential of matrix interference. Acceptance criteria are 85.0 – 115% recovery. Use the recovery data to help evaluate any bias as detailed in the MS/MSD Corrective Action Flowchart (see the Quality Assurance Plan). The LIMS will automatically flag a PDS not meeting the acceptance criteria with a "S" qualifier.

10.11 A *Serial Dilution (SD)* can be analyzed on any sample to evaluate the potential of matrix interference. Acceptance criteria for a 1:5 dilution are ≤ 10% difference between the original and the diluted sample concentration. If acceptance criteria are not met, another serial dilution of a different factor (e.g. 1:10) should be

analyzed. The LIMS will automatically flag a SD not meeting the acceptance criteria with a "R" qualifier.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Perform the required preventative maintenance as necessary.

11.2 If the instrument and autosampler have not been on, turn power on and allow a minimum of 1 hour for the instrument to stabilize. Perform the following steps when the instrument is stable. It is acceptable to leave the instrument and autosampler turned on with the lamp and gas shut off.

11.3 Turn on the lamp and gas. Allow a minimum of 15 minutes for warm up.

11.4 To start the software, double-click on "Tweak232.exe" icon and then double-click on the "Hg Analysis" icon.

11.5 Click on the Worksheet box, then choose Open, click on the high standard, and click OK.

11.6 Go to Instrument, M6000, Controls, and set the gas flow to 175. Click Set Gas to keep this change.

11.7 Go to Auto-sampler page and click the pump on and probe down.

11.8 Click Close to return you to the Analysis page.

11.9 Go to Labels and enter the analytical batch information.

11.10 Go to Rack ID, Sample ID, then right-click. Click on Insert QC in the properties box.

11.11 Scroll to the bottom and click on QC Standard. The number in the first position of the grid should be "1" and the value in the "Insert every nth position" field should be 10. Set these and then click OK.

11.12 Scroll to the bottom and click on QC Blank. The number in the first position of the grid should be "2" and the value in the "Insert every nth position" field should be 11. Set these and then click OK.

11.13 Verify that there are a QC standard and blank set for after every 10 samples and again at the end of the analytical batch.

11.14 Label the initial QC Standard and QC Blank as "ICV" and "ICB", respectively. Label the continuing QC Standards and QC Blanks as "CCV" and "CCB", respectively.

11.15 Clean and rinse the 2L rinse bottle with DI water, refill with 1% $HCl/HNO_3$ solution, and place the autosampler rinse tube into the rinse bottle.

11.16 Inspect the $SnCl_2/HCl$ solution. Replace with fresh solution if it is yellow (oxidized) or precipitated.

11.17 Inspect peristaltic pump tubing and replace if necessary. DO NOT lock the shoe clamps at this time. The 2 waste tubes (cream tubes with yellow bridge stops) are installed at the back of the pump. The sample tube (black) is installed in the third position from the back. The reagent tube (cream tube with black bridge stops) is installed at the front of the pump.

11.18 Place the reagent capillary in a beaker of DI water and start the peristaltic pump (clockwise rotation).

11.19 Lock down the peristaltic pump shoe clamps.

11.20 Inspect the liquid flows. The GLS drain and the waste line from the peristaltic pump should be flowing smoothly without build up or pulsing.

11.21 Wet the center post in the GLS by pinching the drain tube. Hold the tube until 2 or 3 bubbles travel past the center post then release the tube and allow the water level to drain.

11.22 Attach the GLS exhaust tube to the GLS.

11.23 Place reagent capillary in the reagent bottle.

11.24 Go to Instrument and using the autozero function, zero the analyzer.

11.25 To peak profile the instrument, click on Read and the "0" standard at the bottom of the Analysis page. Then, scroll down and click Standards and STD Tube 4 (the high standard). Record the uabs and concentration of the peak profile standard.

11.26 Go to File (on the Analysis page), New Output File to obtain a new page to begin calibration and analysis.

11.27 Again, zero the instrument by going to Instrument, Zero.

11.28 Go to Instrument, Calibrate. The calibration standards (low to high) should be in the first positions. The CCV should be in position 8 and the CCB in position 9. Acceptance criteria is a correlation coefficient of $r \geq 0.995$. If acceptable, continue with sample analysis by clicking Start and Yes (to zero before analyses). If not acceptable, recalibrate (using a New Output File).

## 12.0 PROCEDURE

Sample preparation documentation is maintained using the Digestion Log (copy attached).

12.1 SAMPLE PREPARATION - WATERS

12.1.1 Transfer 25-ml of sample into a digestion tube.

12.1.2 Add 1.25-ml conc. $H_2SO_4$, 1.0-ml conc. $HNO_3$, and 4.0-ml $KMnO_4$ reagent. A purple color should be present. If not, add additional $KMnO_4$ reagent until present.

12.1.3 Allow the sample to set for 15 minutes. If the purple color disappears within that timeframe, add sufficient additional $KMnO_4$ reagent until the color persists for 15 minutes.

12.1.4 Add 2.0-ml $K_2S_2O_8$ reagent.

12.1.5 Mix the sample and digest in a 95°C digestion block for 2 hours. (A water bath must be used for samples applicable to our North Carolina certification, as use of the digestion block is not approved by the NC DENR for Mercury analysis.)

12.1.6 Remove the sample from the digestion block (or water bath) and allow the sample to cool to room temperature.

12.1.7 Add 1.5-ml hydroxylamine reagent, cap the digestion tube and shake to mix. Shake until the purple color disappears.

12.1.8 Dilute the sample to a final volume of 40-ml with DI water.

12.2 SAMPLE PREPARATION - SOLIDS

12.2.1 Transfer 0.6g of sample into a digestion tube and record the weight used. (Use 0.25g of the solid LCS. Use the entire wipe when analyzing environmental wipe samples.)

12.2.2 Add 5.0-ml DI water then 5.0-ml aqua regia.

12.2.3 Mix the sample and digest in a 95°C digestion block for 2 minutes. (A water bath must be used for samples applicable to our North Carolina certification, as use of the digestion block is not approved by the NC DENR for Mercury analysis.)

12.2.4 Remove the sample from the digestion block (or water bath) and allow the samples to cool to room temperature.

12.2.5 Add 15-ml $KMnO_4$ reagent and swirl to mix. A purple color should be present. If not, add additional $KMnO_4$ reagent until present.

12.2.6 Allow the sample to set for 15 minutes. If the purple color disappears within that timeframe, add sufficient additional $KMnO_4$ reagent until the color persists for 15 minutes.

12.2.7 Return the samples to the digestion block (or water bath, as appropriate) and allow digestion at 95°C for 30 minutes.

12.2.8 Remove the sample from the digestion block (or water bath) and allow the sample to cool to room temperature.

12.2.9 Add 6.0-ml hydroxylamine reagent, cap the digestion tube and shake to mix. Shake until the purple color disappears.

12.2.10 Dilute the sample to a final volume of 40-ml with DI water.

12.3 SAMPLE ANALYSIS

Sample analysis documentation is maintained using the instrument software printouts.

12.3.1 Continue with automated analysis as in the Calibration section.

12.4 INSTRUMENT SHUTDOWN

12.4.1 Place the reagent capillary in a beaker of 10% $HNO_3$ and cap the reagent bottle. Allow the system to rinse for a minimum of 10 minutes.

12.4.2 Place the reagent capillary in a beaker of DI water and allow the system to rinse for 1 minute.

12.4.3 Go to Instrument, M6000, Controls, Auto-sampler and click Probe Up and Pump Off to raise the probe.

12.4.4 Remove reagent capillary from DI water.

12.4.5 Allow the drain and waste lines to completely dry.

12.4.6 Turn off the peristaltic pump.

12.4.7 Release the peristaltic pump shoe clamps and pump tubing from the holder clips.

12.4.8 Close vents on waste container.

12.4.9 Remove GLS exhaust line from the GLS.

12.4.10 Turn off the lamp and gas. The instrument may be left powered on if you anticipate its use again within a day or so, otherwise, turn off the instrument and autosampler.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 After review, enter final results into the LIMS system. Results above the linear range of the instrument are flagged with a "Q" on the instrument printout and with a

an "E" qualifier by the LIMS. As there is less certainty in these data, the digestates should be reanalyzed at an appropriate dilution. Details on the procedure for entering analytical data are in the Data Entry SOP.

13.2 The instrument automatically calculates the sample concentration by linear regression comparison with the absorbance data from the calibration curve. The calculation of final concentration is:

$$\text{Hg, ppm} = \frac{[(\text{instrument reading, ug/l})\ (V_f)\ (\text{DF})\ /\ 1000]}{\text{Sample size,-ml or g}}$$

Where: $V_f$ = final volume of digestate
DF = dilution factor

13.3 For wipe samples, enter 1.6 (i.e. 40-ml/25-ml) into the dilution factor (DF) field, and enter the final volume of digestate, in liter units, into the preparation factor (PFac) field.

13.4 The LIMS calculates the dry-weight concentration for solid samples as follows:

$$\text{Conc. Dry} = \frac{(\text{wet weight conc.})}{(100 - \%\text{Moisture})}$$

## 14.0 METHOD PERFORMANCE

14.1 Method Detection Limit

The latest MDL study yielded the following data:

| | | |
|---|---|---|
| n = | 7 | |
| Standard Deviation ($\sigma_{n-1}$) | 0.038 | ug/l |
| Spiked Concentration | 0.20 | ug/l |
| Average Concentration | 0.239 | ug/l |
| Average Recovery | 119 | % |
| Calculated MDL | 0.11 | ug/l |

14.2 Initial Demonstration of Capability

A typical IDC study will yield data similar to:

| | | |
|---|---|---|
| n = | 4 | |
| Standard Deviation ($\sigma_{n-1}$) | 0.022 | ug/l |
| Spiked Concentration | 1.0 | ug/l |
| Average Concentration | 0.88 | ug/l |
| Average Recovery | 88.2 | % |

| | |
|---|---|
| Client: | SIMALABS International |
| SOP ID: | SOP-MET-7041-1 |
| Rev. Number: | 2.0 |
| Rev. Date: | March 1, 1996 |

# Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Antimony For Aqueous Samples

Prepared For SIMALABS International
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7041

Revision # 2.0
Issued: March 1, 1996

| | |
|---|---|
| Immediate Supervisor | Date 3/8/99 |
| Second Supervisor | Date 3/8/99 |
| QA/QC Officer | Date 3/8/99 |
| Analyst | Date 3/8/99 |

Effective: March 1, 1996

**CAUTION**

**Disclaimer: This Standard Operating Procedure has been prepared for the sole use of SIMALABS International and may not be specifically applicable to the activities of other organizations.**

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## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Refer to the SIMALABS International Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

## 17.0 REFERENCES

17.1 USEPA Method 245.1, MCAWW March 1983 and revision 3.0

17.2 SW-846 Methods 7470A and 7471A

17.3 SIMALABS International Quality Assurance Plan, current revision

17.4 SIMALABS Good Laboratory Practices document.

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

Copy of the Digestion Log

# SIMALABS INTERNATIONAL
## MERCURY DIGESTION LOG SHEET

Batch #: _____ Batch ID: _____ Analyst: _____ Peer Check: _____

Hot Plate Temperature: _____ °C Date/Time In: _____ Date/Time Out: _____

Method: 245.1 / 7470A / 7471A

Potassium Permanganate #: _____ Hg ICV / CCV / MS / MSD / LCS #: _____

Potassium Persulfate #: _____ HG Cal Spike #: _____

Hydroxylamine Hydrochloride #: _____ Solid LCS #: _____

| | Simalabs I.D. | Container # | mL or g | MS | MSD | mL Final Vol | Comments |
|---|---|---|---|---|---|---|---|
| BLK | | | | | | | |
| LCS | | | | | | | |
| 1 | | | | | | | |
| 2 | | | | | | | |
| 3 | | | | | | | |
| 4 | | | | | | | |
| 5 | | | | | | | |
| 6 | | | | | | | |
| 7 | | | | | | | |
| 8 | | | | | | | |
| 9 | | | | | | | |
| 10 | | | | | | | |
| 11 | | | | | | | |
| 12 | | | | | | | |
| 13 | | | | | | | |
| 14 | | | | | | | |
| 15 | | | | | | | |
| 16 | | | | | | | |
| 17 | | | | | | | |
| 18 | | | | | | | |
| 19 | | | | | | | |
| 20 | | | | | | | |

revision: b_8-01

$HNO_3$ Lot #: _____ HCl Lot #: _____ $H_2SO_4$ Lot #: _____

Received from prep by: _____

STANDARD OPERATING PROCEDURE

# GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF ANTIMONY FOR AQUEOUS SAMPLES

## LOCATION:

Metals, Metals Laboratory

## REFERENCE:

SW-846, 3rd Edition, Method 7041

## MATRIX:

Water, Leachates

## QUANTITATION LIMIT:

EQL = 50 ug/L; MDL = 11.5 ug/L.

## RANGE:

50 ug/L to 200 ug/L without dilution

## PRINCIPLE, SCOPE, AND APPLICATION:

Antimony in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using

furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

## INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

(1) Successively dilute and reanalyze the samples to eliminate interferences.

(2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-bank absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for,

chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilzation step. If another acid in addition to $HNO_3$ is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

## SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during digestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestions are to occur under a fume hood.

## SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous and leachate samples are to be collected in 500 ml plastic containers with teflon lined lids, preserved to pH $\leq$ 2 with nitric acid, and cooled to 4°C until digestion. Samples must be analyzed within 6 months of collection.

## APPARATUS:

1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.

2) Zeeman Graphite Tube Atomizer provides power to furnace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 °C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/min.

3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 ul.

4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and provides data manipulation and reporting of sample calculations.

5) Citizen dot matrix printer, prints calibration and sample results.

6) Class A volumetric pipets

7) Class A volumetric flasks

8) Pipets: Microliter, with disposable tips. sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible sources of contamination prior to their use.

9) Analytical balance

10) Disposable glass serological pipets

ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Furnace optics are cleaned twice weekly. Plumbing connections , and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

REAGENTS AND CALIBRATION STANDARDS:

1) Deionized water - Type II

2) Nitric Acid - concentrated, trace metals grade (Fisher, AS09-212)

3) Furnace stock calibration standard: Using a Class A volumetric pipet, dilute 1.0 ml Cadmium Stock (Spex, PLSB7-2y, 1000 ppm), and 4.0 ml concentrated nitric acid, to 200 ml with DI water in a volumetric flask. Bring to volume. This will result in a final concentration of 10 ppm Antimony. Dilute stock calibration standard 1:99 with DI water for daily calibration.

4) Furnace ICV/CCV Solutions: Using a 100 uL micropipet and a 10 mL glass serological pipet, transfer 0.10 ml QC-19 Stock (SPEX , QC-19- 100 ppm) and 2.0 ml concentrated nitric acid to a 100 ml Class A volumetric flask partially filled with DI water. Bring to volume. This will result in a 100 ppb final concentration.

5) Antimony Modifier: Using an analytical balance, weigh 3.00 g $NH_4NO_3$ (Fisher, A684-500) and transfer to a 100 mL Class A volumetric flask. Dilute to 100 ml with DI water in a volumetric flask.

## CALIBRATION PROCEDURES:

A curve consisting of 4 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of $\geq$ 0.995 to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the PQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCV and CCB must meet the above stated criteria for the ICV and ICB.

## SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

## ANALYSIS PROCEDURE:

1.) Turn on monitor, computer, Spectra AA-400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press

F10 (index) on computer keyboard. Type 10, press F-6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.

2.) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swap and isopropyl rubbing alcohol. Clean a graphite tube and it's platform using a Kim wipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite tube to the center of the furnace chimney. Swing the toggle lever counter-clockwise in order to close the right-hand housing onto the tube now positioned inside the furnace housing.

3.) Remove rinse bottle and fill to the line with DI $H_2O$. Clean the Blank, Modifier and Standard cups with DI $H_2O$ and 1:1 Nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.

4.) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampling arm will move from it's rinse position to the sample 1 position and than to the sample introduction hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. Use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5.) Open syringe compartment door. Put the syringe clear of it's mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Re-insert the syringe assembly into it's housing and close the compartment door.

6.) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean). The furnace will heat and clean the graphite tube. Press shift and F11 (start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is properly injected onto the platform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it in the path of the UV light

and thus putting in view the position of the capillary while inside the graphic tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the analytical signal.

7.) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knobs on the left-hand side of the appropriate lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its furthest most position from the left-hand baseline.) Note: pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8.) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour check standards into sample cups and place them in their proper positions in the autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for Antimony:

Antimony: Program #8, Matrix Modifier - Antimony Modifier
Standard 1 =50 ppb, 2 = 100 ppb, 3 = 150 ppb, 4 = 200 ppb
ICV = 100 ppb, CCV = 100 ppb

QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within $\pm$ 20% of true value.

At least one preparatory blank, laboratory standard, spike and one duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See below.)

*Method of standard additions:*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

1) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

3) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume $V_x$, are taken. To the first (labeled A) is added a small volume $V_s$ of a standard analyte solution of concentrate $c_s$. To the second (labeled B) is added the same volume $V_s$ of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration $c_x$ is calculated:

$$c_x = \frac{S_B V_S c_S}{(S_A - S_B) V_X}$$

where,

$S_A$ and $S_B$ are the analytical signals (corrected for the blank) of solutions A and B, respectively. $V_S$ and $c_s$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_S$ is made much less than $V_X$, and thus $c_s$ is much greater than $c_x$, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

## DATA TREATMENT:

For determination of metal concentration by direct aspiration and furnace; read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

$$\text{ug/L metal in sample} = A \times \frac{(C + B)}{C}$$

where,

A = ug/L of metal in diluted aliquot from calibration curve
B = Acid blank matrix used for dilution, mL
C = sample aliquot, mL

## DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix

- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration Determined and resulting PQL
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- Spike Sample Recovery form
- Laboratory Control Sample Summary form
- All Raw Data
- Preparation Records

| | |
|---|---|
| Client: | IDEM-~~BAA 95-30~~ MET-20-97 |
| SOP ID: | SOP-MET-7131-1 |
| Rev. Number: | 2.0 |
| Rev. Date: | March 1, 1996 |

# Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Cadmium For Nonaqueous Samples

Author: Karin Stewart
Prepared For American Analytical, Inc.
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7131

Revision # 2.0
Issued: March 1, 1996

| | |
|---|---|
| Immediate Supervisor | Date 1-19-97 |
| Second Supervisor | Date 1/15/97 |
| QA/QC Officer | Date 1-19-97 |
| Analyst | Date 1-14-97 |

Effective: March 1, 1996

CAUTION

<u>Disclaimer:</u> **This Standard Operating Procedure has been prepared for the sole use of American Analytical, Inc. and may not be specifically applicable to the activities of other organizations.**

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# STANDARD OPERATING PROCEDURE
# GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF CADMIUM FOR NONAQUEOUS SAMPLES

## LOCATION:

Metals, Metals Laboratory

## REFERENCE:

SW-846, 3rd Edition, Method 7131

## MATRIX:

Soils, solids, sludges

## QUANTITATION LIMIT:

EQL = 200 ug/kg.

## RANGE:

200 ug/kg to 2000 ug/kg without dilution

## PRINCIPLE, SCOPE, AND APPLICATION:

Cadmium in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

(1) Successively dilute and reanalyze the samples to eliminate interferences.

(2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-bank absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way,

broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilzation step. If another acid in addition to $HNO_3$ is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

## SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during digestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestions are to occur under a fume hood.

## SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Nonaqueous samples are to be collected in 4 oz. squat jars with teflon lined lids, and cooled to 4°C until digestion. Samples must be analyzed within 6 months of collection.

## APPARATUS:

1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.

2) Zeeman Graphite Tube Atomizer provides power to furnace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 °C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/min.

3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 ul.

4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and

provides data manipulation and reporting of sample calculations.

5) Citizen dot matrix printer, prints calibration and sample results.

6) Class A volumetric pipets

7) Class A volumetric flasks

8) Pipets: Microliter, with disposable tips. sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible sources of contamination prior to their use.

9) Analytical balance

10) Disposable glass serological pipets

ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Furnace optics are cleaned twice weekly. Plumbing connections , and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

REAGENTS AND CALIBRATION STANDARDS:

1) Deionized water - Type II

2) Nitric Acid - concentrated, trace metals grade (Fisher, AS09-212)

3) Furnace stock calibration standard: Using a Class A volumetric pipet, dilute 1.0 ml Cadmium Stock (Spex, QC-19, 1000 ppm), and 4.0 ml concentrated nitric acid, to 200 ml with DI water in a volumetric flask. Bring to volume. This will result in a final concentration of 10 ppm Cadmium. Dilute stock calibration standard 1:99 with DI water for daily calibration.

4) Furnace ICV/CCV Solution: Using a 100 uL micropipet and a 10 mL disposable glass serological pipet, transfer 0.10 ml QC-19 Stock (SPEX , QC-19, 100 ppm) and 2.0 ml concentrated nitric acid to a 100 ml Class A volumetric flask partially filled with DI water. Bring to volume. This will result in a 100 ppb final concentration. Dilute 1:1 for a working concentration of 50 ppb.

5) Cadmium Modifier: Using an analytical balance, weigh 1.00 g $NH_4H_2PO_4$ (Fisher, A684-500) and transfer to a 100 mL Class A volumetric flask. Dilute to volume.

## CALIBRATION PROCEDURES:

A curve consisting of 4 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of $\geq$ 0.995 to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the PQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCV and CCB must meet the above stated criteria for the ICV and ICB.

## SAMPLE PREPARATION:

Nonaqueous: See SOP-MET-3050-1

## ANALYSIS PROCEDURE:

1.) Turn on monitor, computer, Spectra AA-400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F-6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.

2.) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swap and isopropyl rubbing alcohol. Clean a graphite tube and it's platform using a Kim wipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite tube to the center of the furnace chimney. Swing the toggle lever counter-clockwise in order to close the right-hand housing onto the tube now positioned inside the furnace housing.

3.) Remove rinse bottle and fill to the line with DI $H_2O$. Clean the Blank, Modifier and Standard cups with DI $H_2O$ and 1:1 Nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.

4.) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampling arm will move from it's rinse position to the sample 1 position and than to the sample introduction hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. Use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5.) Open syringe compartment door. Put the syringe clear of it's mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Re-insert the syringe assembly into it's housing and close the compartment door.

6.) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean). The furnace will heat and clean the graphite tube. Press shift and F11 (start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is properly injected onto the platform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it in the path of the UV light and thus putting in view the position of the capillary while inside the graphic tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the analytical signal.

7.) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knobs on the left-hand side of the appropriate lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its furthest most position from the left-hand baseline.) Note: pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8.) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour check standards into sample cups and place them in their proper positions in the autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for Cadmium:

Cadmium: Program #6, Matrix Modifier - Cadmium Modifier
Standard 1 =0.25 ppb, 2 = 0.50 ppb, 3 = 1.00 ppb, 4 = 2.50 ppb
ICV = 1.00 ppb, CCV = 1.00 ppb

QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within ± 20% of true value.

At least one preparatory blank, laboratory standard, spike and duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See below.)

*Method of standard additions:*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

1) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

3) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume $V_X$, are taken. To the first (labeled A) is added a small volume $V_S$ of a standard analyte solution of concentrate $c_S$. To the second (labeled B) is added the same volume $V_S$ of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration $c_X$ is calculated:

$$c_X = \frac{S_B V_S c_S}{(S_A - S_B) V_X}$$

where,

$S_A$ and $S_B$ are the analytical signals (corrected for the blank) of solutions A and B, respectively. $V_S$ and $c_S$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_S$ is made much less than $V_X$, and thus $c_S$ is much greater than $c_X$, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

## DATA TREATMENT:

For determination of metal concentration by direct aspiration and furnace; read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

$$\text{ug/L metal in sample} = A \times \frac{(C + B)}{C}$$

where,

A = ug/L of metal in diluted aliquot from calibration curve
B = Acid blank matrix used for dilution, mL
C = Sample aliquot, mL

For nonaqueous samples:

ug/kg metal in sample = (A)(V)/(W)
where,

A = ug/L of metal in processed sample from calibration curve
V = Final volume of the processed
W = Weight of sample, grams

DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration Determined and resulting PQL
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- Spike Sample Recovery form
- Laboratory Control Sample Summary form
- All Raw Data
- Preparation Records

# Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Lead For Aqueous Samples

Prepared For SIMALABS International
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7421

Revision # 2.0
Issued: March 5, 1999

| | |
|---|---|
| Immediate Supervisor | Date 3/8/99 |
| Second Supervisor | Date 3/8/99 |
| QA/QC Officer | Date 3/8/99 |
| Analyst | Date 3/8/99 |

Effective: March 1, 1996

**CAUTION**

**Disclaimer: This Standard Operating Procedure has been prepared for the sole use of SIMALABS International and may not be specifically applicable to the activities of other organizations.**

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# STANDARD OPERATING PROCEDURE
# GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF LEAD FOR AQUEOUS SAMPLES

## LOCATION:

Metals, Metals Laboratory

## REFERENCE:

SW-846, 3rd Edition, Method 7421

## MATRIX:

Water, Leachate

## DETECTION LIMITS:

EQL = 5.0 ug/ L; MDL = 0.499 ug/ L

## RANGE:

5.0 ug L to 50 ug L without dilution

## PRINCIPLE, SCOPE AND APPLICATION:

Lead in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the mati<rices and models of atomic absorption spectrophotometers. When using

furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.
When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a reqresentative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected speciment to be volatilized. A monochromator isolates the dishcarge lamp, and a photosensitive device measures the attenuated transmitted radation.

## INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

1) Successively dilute and reanalyze the samples to eliminate interferences.

2) Analyze the sample by method of standard addditions while noticing the precautions and limitations of its use.

Gasas generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-bank absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometines be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilzation step. If another acid in addition to HNO3 is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If contamination is suspected , the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

## SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during degestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestion are to occur under a fume hood. CAUTION: Lead isextremely toxic! Handle with care. Refer to MSDS for any inquiries about reagents or chemicals used in this test.

## SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous and leachate samples are to be collected in 500 mL plastic containers with teflon lined lids, and preserved to pH $\leq$ 2 with nitric acid. Samples should be labeled as being preserved. Samples must be analyzed within 6 months of collection. Samples will be rejected if container is cracked or broken, at which time the immediate supervisor will be notified.

## APPARATUS:

1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.

2) Zeeman Graphte Tube Atomizer provides power to furnace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/ min.

3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 uL.

4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and provides data manipulation and reporting of samples calculaitons.

5) Citizen dot matrix printer, prints calibration and sample results.

6) Class A volumetric flasks

8) Pipets: Microliter, with disposable tips. Sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible source of contamination prior to their use.

9) Class A volumetric pipets

10) Analytical balance

11) Disposable glass serological pipets

ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Frunace optics are cleaned twice weekly. Plumbing connections, and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

REAGENTS AND CALIBRATION STANDARDS:

1) Deionized water - Type II

2) Nitric Acid - concentrated, trace metals grade (Fisher, AS09-212)

3) Furnace Stock Calibration Standard: Using a Class A volumetric pipet, dilute 2.0 mL lead stock (Spex, PLB2-2y), and 4.0 mL concentrated nitric acid, to 200 mL with DI water in a volumetric flask. This will result in a final concentration of 10 ppm lead. Dilute stock calibration standard 1:99 with DI water for daily calibration.

4) Furnace ICV/CCV Solutions: Using a 100 uL micropipet and a 10 mL disposable glass serological pipet, transfer 0.10 mL QC-19 stock (Spex, QC-19, 100 ppm) and 2.0 mL concentrated nitric acid to a 100 mL Class A volumetric flask. Bring to volume. This will result in a 100 ppb final concentration. Dilute 1:1 for a working concentration of 50 ppb.

5) Lead Modifier: Using a Class A volumetric pipet, transfer 1.00 mL phosphoric acid into a Class A volumetric flask, partially filled with DI water. Bring to volume.

## CALIBRATION PROCEDURES:

A curve consisting of 5 standards and a blank is analyzed at the beginning of each run The curve must demonstrate a correlation coefficient of $\geq$ 0.995 to be valid. An ICV followed by an ICB are analyzed prior to sample anlaysis. the ICV must recover within 20 % of true value, and the ICB must show results less than the EQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCB and CCV must meet the above stated criteria for the ICV and ICB.

## SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

## ANALYSIS PROCEDURE:

1) Turn on monitor, computer, Spectra AA 400, Zeeman, Graphic Tube Atomizer,

T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F6 (new page) press F1 (clear sequence).

Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.

2) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swab and isopropyl rubbing alcohol. Clean a graphite tube and its plateform using a Kimwipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite tube to the center of the furnace dhimney. Swing the toggle lever counter-clockwise in order to close the righthand housing onto the tube now positioned inside the furnace housing.

3) Remove rinse bottle and fill to the line with DI H2O. Clean the blank, modifier, and standard cups with DI H2O and 1:1 nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.

4) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampleing arm will move from its rinse position to the sample 1 position and then to the sample introductrion hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5) Open syringe compartment door. Put the syringe clear of its mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Reinsert the syringe assembly into its housing and close the compartment door.

6) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean).
The furnace will heat and clean the graphite tube. Press shift and F11 ( start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is properly injected onto the plateform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it
in the path of the UV light and thus putting in view the position of the capillary while inside the graphic tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the anlaytical
signal.

7) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knmobs on the left-hand side of the appropriate
lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its furthest most position from the left-hand baseline). Note:
pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour
check standards into sample cups and place them in their proper positions in the
autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for lead:

Lead: Program #4, Matrix Modifier - Lead Modifier
Standard 1 = 5.00 ppb, 2 = 10.0 ppb, 3 = 25.0 ppb, 4 = 50.0 ppb, 5 = 99.00 ppb
ICV = 50.0 ppb, CCV = 50.0 ppb

QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within ± 20% of true value.

At least one preparatory blank, laboratory standard, spike, and duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See below).

*Method of standard additions*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is sa\caled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

1) The absorbance plot of sample and standards must be linear over the concentration range of concern. for best results, the slope of the plost should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be used.

2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changesk, and the standard addition should

respond in a similar manner as the anlayte.

3) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single addition method, in which two identical aliquots of the sample solution, each of Volume $V_x$ are taken. To the first (labeled A) is added a small valume $V_s$ of a standard analyte solution of concentrate $c_s$. to the second (labeled B) is added the same volume $V_s$ of the solvent. The analytical signals of A and B are measured and corrected for nonanlyte signals. The unknown sample concentration $c_x$ is calculated:

$$c_x = S_b V_s C_s / (S_a - S_b) V_x$$

where,

$S_a$ and $S_b$ are the analytical signals (corrected for the blank) of solutions a and b respectively. $V_s$ and $c_s$ should be chosen so that $S_a$ is roughly twice $S_b$ on the average. It is best if $V_s$ is made much less than $V_x$ and thus $c_s$ is much greater than $c_x$ to avoid excess dilution of the sample matrix. It a searation or concentration step is used, the additions are best made first and carried throught the entire procedure.

DATA TREATMENT:

For determination of metal concentration by direct aspiration and furnace, read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

ug/L metal in sample = (A) [ (C + B)/C ]

where,

A = ug/L of metal in diluted aliquot from calibration curve

B = acid blank matrix used for dilution, mL
C = sample aliquot, mL

DATA DELIVERABLES:

Reports to clients will include:

-Date of receipt
-Date of preparation
-Date of analysis
-Analyst
-Matrix
-Laboratory ID#
-Client ID#
-Analytical method #
-Concentration determined and resulting EQL
-ICV, CCV, summary form
-ICB, CCB, prep blank summary form
-Spike sample recovery form
-Laboratory control sample summary form
-All raw data
-Preparation records

| | |
|---|---|
| Client: | SIMALABS International |
| SOP ID: | SOP-MET-7060-1 |
| Rev. Number: | 2.0 |
| Rev. Date: | March 1, 1996 |

# Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Arsenic For Aqueous Samples

Prepared For SIMALABS International
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7060

Revision # 2.0
Issued: March 1, 1996

| | |
|---|---|
| Immediate Supervisor | Date 3/8/99 |
| Second Supervisor | Date 3/8/99 |
| QA/QC Officer | Date 3/8/99 |
| Analyst | Date 3/8/99 |

Effective: March 1, 1996

**CAUTION**

**Disclaimer: This Standard Operating Procedure has been prepared for the sole use of SIMALABS International and may not be specifically applicable to the activities of other organizations.**

# TABLE OF CONTENTS

# STANDARD OPERATING PROCEDURE
# GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF ARSENIC FOR AQUEOUS SAMPLES

## LOCATION:

Metals, Metals Laboratory

## REFERENCE:

SW-846, 3rd Edition, Method 7060

## MATRIX:

Water, Leachate

## DETECTION LIMIT:

EQL = 10 ug/L; MDL = 2.6 ug/L

## RANGE:

10 ug/L to 100 ug/L without dilution

## PRINCIPLE, SCOPE, AND APPLICATION:

Arsenic in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using furnace techniques the analyst should be cautioned as to possible chemical

reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

(1) Successively dilute and reanalyze the samples to eliminate interferences.

(2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-bank absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for,

chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilzation step. If another acid in addition to $HNO_3$ is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

## SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during digestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestions are to occur under a fume hood.

## SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous and leachate samples are to be collected in 500 ml plastic containers with teflon lined lids, and preserved to $pH \leq 2$ with nitric acid, and cooled to 4°C until digestion. Samples must be analyzed within 6 months of collection.

## APPARATUS:

1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.

2) Zeeman Graphite Tube Atomizer provides power to furnace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 °C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/min.

3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 ul.

4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and provides data manipulation and reporting of sample calculations.

5) Citizen dot matrix printer, prints calibration and sample results.

6) Class A volumetric pipets

7) Class A volumetric flasks

8) Pipets: Microliter, with disposable tips. sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible sources of contamination prior to their use.

9) Analytical balance

10) Disposable galss serological pipets

## ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Furnace optics are cleaned twice weekly. Plumbing connections , and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

## REAGENTS AND CALIBRATION STANDARDS:

1) Deionized water - Type II

2) Nitric Acid - concentrated, trace metals grade (Fisher, AS09-212)

3) Furnace stock calibration standard: Using a Class A volumetric pipet, dilute 2.0 ml Arsenic Stock (Spex, PLAS2-2y), and 4.0 ml concentrated nitric acid, to 200 ml with DI water in a Class A volumetric flask. This will result in a final concentration of 10 ppm Arsenic. Dilute stock calibration standard 1:99 with DI water for daily calibration.

4) Furnace ICV/CCV Solutions: Using a 100 uL micropipet and a 10 mL glass serological pipet, transfer 0.10 ml QC-19 Stock (SPEX, QC-19, 100 ppm) and 2.0 mL concentrated nitric acid to a100 ml Class A volumetric flask partially filled with DI water. Bring to volume. This will result in a 100 ppb final concentration. Dilute 1:1 for a working concentration of 50 ppb.

5) Nickel Nitrate Modifier: Using an analytical balance, weigh 0.4950 g $Ni(NO_3)*6H_2O$ (Malinckrodt, UN2725). Using a 10 mL glass disposable serological pipet, transfer 5.0 ml concentrated nitric acid into a Class A volumetric flask partially filled with DI water. Transfer weighed $Ni(NO_3)*6\ H_2O$ into the flask. Bring to volume and mix until dissolved.

## CALIBRATION PROCEDURES:

A curve consisting of 4 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of $\geq$ 0.995 to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the EQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCV and CCB must meet the above stated criteria for the ICV and ICB.

## SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

## ANALYSIS PROCEDURE:

1.) Turn on monitor, computer, Spectra AA-400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F-6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.

2.) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swap and isopropyl rubbing alcohol. Clean a graphite tube and it's platform using a Kim wipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite tube to the center of the furnace chimney. Swing the toggle lever counter-clockwise in order to close the right-hand housing onto the tube now positioned inside the furnace housing.

3.) Remove rinse bottle and fill to the line with DI $H_2O$. Clean the Blank, Modifier and Standard cups with DI $H_2O$ and 1:1 Nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.

4.) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampling arm will move from it's rinse position to the sample 1 position and than to the sample introduction hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. Use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5.) Open syringe compartment door. Put the syringe clear of it's mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Re-insert the syringe assembly into it's housing and close the compartment door.

6.) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean). The furnace will heat and clean the graphite tube. Press shift and F11 (start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is

properly injected onto the platform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it in the path of the UV light and thus putting in view the position of the capillary while inside the graphic tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the analytical signal.

7.) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knobs on the left-hand side of the appropriate lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its furthest most position from the left-hand baseline.) Note: pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8.) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour check standards into sample cups and place them in their proper positions in the autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for Arsenic:

Arsenic: Program #2, Matrix Modifier - Nickel Nitrate
Standard 1 = 10ppb, 2 = 25ppb, 3 = 50ppb, 4 = 100ppb
ICV = 50ppb, CCV = 50ppb

QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within $\pm$ 20% of true value.

At least one preparatory blank, laboratory standard, spike and duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See below.)

*Method of standard additions:*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

(a) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

(b) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

(c) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume $V_x$, are taken. To the first (labeled A) is added a small volume $V_s$ of a standard analyte solution of concentrate $c_s$. To the second (labeled B) is added the same volume $V_s$ of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration $c_x$ is calculated:

$$c_x = \frac{S_B V_S c_S}{(S_A - S_B) V_X}$$

where,

$S_A$ and $S_B$ are the analytical signals (corrected for the blank) of solutions A and B, respectively. $V_S$ and $c_s$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_S$ is made much less than $V_X$, and thus $c_s$ is much greater than $c_x$, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

## DATA TREATMENT:

For determination of metal concentration by direct aspiration and furnace; read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

$$\text{ug/L metal in sample} = A \times \frac{(C + B)}{C}$$

where,

A = ug/L of metal in diluted aliquot from calibration curve

B = Acid blank matrix used for dilution, mL

C = sample aliquot, mL

## DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix

- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration Determined and resulting EQL
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- Spike Sample Recovery form
- Laboratory Control Sample Summary form
- All Raw Data
- Preparation Records

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|---|---|
| Client: | SIMALABS INTERNATIONAL |
| SOP ID: | SOP-MET-7740-1 |
| Rev. Number: | 2.0 |
| Rev. Date: | March 1, 1996 |

# Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Selenium For Aqueous Samples

Prepared For SimaLabs International
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7740

Revision # 2.0
Issued: March 1, 1996

| | |
|---|---|
| Immediate Supervisor | Date 3/8/99 |
| Second Supervisor | Date 3/8/99 |
| QA/QC Officer | Date 3/8/99 |
| Analyst | Date 3/8/99 |

Effective: March 1, 1996

**CAUTION**

**Disclaimer: This Standard Operating Procedure has been prepared for the sole use of SimaLabs International and may not be specifically applicable to the activities of other organizations.**

# TABLE OF CONTENTS

# STANDARD OPERATING PROCEDURE
# GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF SELENIUM FOR AQUEOUS SAMPLES

## LOCATION:

Metals, Metals Laboratory

## REFERENCE:

SW-846, 3rd Edition, Method 7740

## MATRIX:

Water, Leachate

## DETECTION LIMIT:

EQL = 5 ug/L; MDL = 1.6 ug/L

## RANGE:

5.00 ug/L to 50.0 ug/L without dilution

## PRINCIPLE, SCOPE, AND APPLICATION:

Selenium in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either

suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

## INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

(1) Successively dilute and reanalyze the samples to eliminate interferences.

(2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-bank absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilzation step. If another acid in addition to $HNO_3$ is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

## SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during digestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestions are to occur under a fume hood.

## SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous samples are to be collected in 500 mL plastic containers with teflon lined lids, preserved to pH $\leq$ 2 with nitric acid, and cooled to 4°C until digestion. Samples must be analyzed within 6 months of collection.

## APPARATUS:

1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.

2) Zeeman Graphite Tube Atomizer provides power to furnace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 °C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/min.

3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 ul.

4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and provides data manipulation and reporting of sample calculations.

5) Citizen dot matrix printer, prints calibration and sample results.

6) Class A volumetric pipets

7) Class A volumetric flasks

8) Pipets: Microliter, with disposable tips. sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible sources of contamination prior to their use.

9) Analytical balance

10) Disposable glass serological pipets

## ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Furnace optics are cleaned twice weekly. Plumbing connections , and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

## REAGENTS AND CALIBRATION STANDARDS:

1) Deionized water - Type II

2) Nitric Acid - concentrated, trace metals grade (Fisher, AS09-212)

3) Furnace stock calibration standard: Using a Class A volumetric pipet, dilute 1.0 ml Selenium Stock (Spex, PLSE2-2y - 100 ppm), and 4.0 ml concentrated

nitric acid, to 200 ml with DI water in a volumetric flask. This will result in a final concentration of 5 ppm Selenium. Dilute stock calibration standard 1:99 with DI water for daily calibration.

4) Furnace ICV/CCV Solutions: Using a 100 uL micropipet and a 10 mL glass serological disposable pipet, transfer 0.10 ml QC-19 Stock (SPEX , QC-19, 100 ppm) and 2.0 ml concentrated nitric acid to 100 ml with DI water in a volumetric flask. This will result in a 100 ppb final concentration. Dilute 1:3 for a working concentration of 25 ppb.

5) Nickel Nitrate Modifier: Using an analytical balance, weigh 0.4950 g $Ni(NO_3)*6H_2O$ (Malinckrodt, UN2725). Using a 10 mL glass disposable serological pipet, transfer 5.0 ml concentrated nitric acid into a Class A volumetric flask partially filled with DI water. Transfer weighed $Ni(NO_3) * 6 H_2O$ into the flask. Bring to volume and mix until dissolved.

## CALIBRATION PROCEDURES:

A curve consisting of 4 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of $\geq$ 0.995 to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the EQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCV and CCB must meet the above stated criteria for the ICV and ICB.

## SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

## ANALYSIS PROCEDURE:

1.) Turn on monitor, computer, Spectra AA-400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F-6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.

2.) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swap and isopropyl rubbing alcohol. Clean a graphite tube and it's platform using a Kim wipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite tube to the center of the furnace chimney. Swing the toggle lever counter-clockwise in order to close the right-hand housing onto the tube now positioned inside the furnace housing.

3.) Remove rinse bottle and fill to the line with DI $H_2O$. Clean the Blank, Modifier and Standard cups with DI $H_2O$ and 1:1 Nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.

4.) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampling arm will move from it's rinse position to the sample 1 position and than to the sample introduction hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. Use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5.) Open syringe compartment door. Put the syringe clear of it's mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Re-insert the syringe assembly into it's housing and close the compartment door.

6.) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean). The furnace will heat and clean the graphite tube. Press shift and F11 (start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is properly injected onto the platform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it in the path of the UV light and thus putting in view the position of the capillary while inside the graphic tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the analytical signal.

7.) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knobs on the left-hand side of the appropriate lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its furthest most position from the left-hand baseline.) Note: pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8.) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour check standards into sample cups and place them in their proper positions in the autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for Selenium:

Selenium: Program #10, Matrix Modifier - Nickel Nitrate
Standard 1 = 5ppb, 2 = 10ppb, 3 = 25ppb, 4 = 50ppb
ICV = 25ppb, CCV = 25ppb

QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within $\pm$ 20% of true value.

At least one preparatory blank, laboratory standard, spike and duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See below.)

*Method of standard additions:*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

(a) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

(b) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

(c) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume $V_x$, are taken. To the first (labeled A) is added a small volume $V_s$ of a standard analyte solution of concentrate $c_s$. To the second (labeled B) is added the same volume $V_s$ of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration $c_x$ is calculated:

$$c_x = \frac{S_B V_S c_S}{(S_A - S_B) V_X}$$

where,

$S_A$ and $S_B$ are the analytical signals (corrected for the blank) of solutions A and B, respectively. $V_S$ and $c_s$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_S$ is made much less than $V_X$, and thus $c_s$ is much greater than $c_x$, to avoid excess dilution of the sample matrix. If a separation or

concentration step is used, the additions are best made first and carried through the entire procedure.

## DATA TREATMENT:

For determination of metal concentration by direct aspiration and furnace; read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

$$\text{ug/L metal in sample} = A \times \frac{(C + B)}{C}$$

where,

A = ug/L of metal in diluted aliquot from calibration curve.
B = Acid blank matrix used for dilution, mL
C = Sample aliquot, mL

## DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration Determined and resulting EQL
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- Spike Sample Recovery form
- Laboratory Control Sample Summary form
- All Raw Data
- Preparation Records

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| Client: | SIMALABS International |
| SOP ID: | SOP-MET-7841-1 |
| Rev. Number: | 2.0 |
| Rev. Date: | March 1, 1996 |

# Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Thallium For Aqueous Samples

Prepared For SIMALABS International
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7841

Revision # 2.0
Issued: March 1, 1996

| | |
|---|---|
| Immediate Supervisor | Date 3/8/99 |
| Second Supervisor | Date 3/8/99 |
| QA/QC Officer | Date 3/8/99 |
| Analyst | Date 3/8/99 |

Effective: March 1, 1996

**CAUTION**

**Disclaimer: This Standard Operating Procedure has been prepared for the sole use of SIMALABS International and may not be specifically applicable to the activities of other organizations.**

# TABLE OF CONTENTS

# STANDARD OPERATING PROCEDURE
# GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF THALLIUM FOR AQUEOUS SAMPLES

## LOCATION:

Metals, Metals Laboratory

## REFERENCE:

SW-846, 3rd Edition, Method 7841

## MATRIX:

Water, Leachate

## DETECTION LIMIT:

EQL = 5 ug/L; MDL = 0.71 ug/L

## RANGE:

5 ug/L to 50 ug/L without dilution

## PRINCIPLE, SCOPE, AND APPLICATION:

Thallium in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

(1) Successively dilute and reanalyze the samples to eliminate interferences.

(2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-bank absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilzation step. If another acid in addition to $HNO_3$ is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during digestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestions are to occur under a fume hood.

## SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous and leachate samples are to be collected in 500 ml plastic containers with teflon lined lids, and preserved to pH $\leq$ 2 with nitric acid, and cooled to 4°C until digestion. Samples must be analyzed within 6 months of collection.

## APPARATUS:

1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.

2) Zeeman Graphite Tube Atomizer provides power to furnace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 °C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/min.

3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 ul.

4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and provides data manipulation and reporting of sample calculations.

5) Citizen dot matrix printer, prints calibration and sample results.

6) Class A volumetric pipets

7) Class A volumetric flasks

8) Pipets: Microliter, with disposable tips. sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible sources of contamination prior to their use.

9) Analytical balance

10) Disposable glass serological pipets

## ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Furnace optics are cleaned twice weekly. Plumbing connections , and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

REAGENTS AND CALIBRATION STANDARDS:

1) Deionized water - Type II

2) Nitric Acid - concentrated, trace metals grade (FISHER AS09-212).

3) Furnace stock calibration standard: Using a Class A volumetric pipet, dilute 1.0 ml Thallium Stock (Spex, QC-19 - 100 ppm), and 4.0 ml concentrated nitric acid, to 200 ml with DI water in a Class A volumetric flask. This will result in a final concentration of 5 ppm Thallium. Dilute stock calibration standard 1:99 with DI water for daily calibration.

4) Furnace ICV/CCV Solutions: Using a 100 uL micropipet and a 10 mL glass serological pipet, transfer 0.10 ml QC-19 Stock (SPEX - QC-19, 100 ppm) and 2.0 ml concentrated nitric acid to a 100 ml Class A volumetric flask partially filled with DI water. Bring to volume. Dilute this solution 1:3, which will result in a 25 ppb final concentration.

5) Citric Acid & Palladium Modifier: Using an analytical balance, weigh 5.00g Citric Acid. (Fisher, A104-500) and transfer to a 100 mL Class A volumetric flask. Dilute to volume. Combine with 100 ml of 1000 ppm Palladium stock (Fisher, PLPD3-2y).

CALIBRATION PROCEDURES:

A curve consisting of 4 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of $\geq$ 0.995 to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the EQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCV and CCB must meet the above stated criteria for the ICV and ICB.

SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

ANALYSIS PROCEDURE:

1.) Turn on monitor, computer, Spectra AA-400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F-6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.

2.) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swap and isopropyl rubbing alcohol. Clean a graphite tube and it's platform using a Kim wipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite

tube to the center of the furnace chimney. Swing the toggle lever counter-clockwise in order to close the right-hand housing onto the tube now positioned inside the furnace housing.

3.) Remove rinse bottle and fill to the line with DI $H_2O$. Clean the Blank, Modifier and Standard cups with DI $H_2O$ and 1:1 Nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.

4.) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampling arm will move from it's rinse position to the sample 1 position and than to the sample introduction hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. Use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5.) Open syringe compartment door. Put the syringe clear of it's mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Re-insert the syringe assembly into it's housing and close the compartment door.

6.) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean). The furnace will heat and clean the graphite tube. Press shift and F11 (start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is properly injected onto the platform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it in the path of the UV light and thus putting in view the position of the capillary while inside the graphic tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the analytical signal.

7.) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knobs on the left-hand side of the appropriate lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its furthest most position from the left-hand baseline.) Note: pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8.) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour check standards into sample cups and place them in their proper positions in the autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for Thallium:

Thallium: Program #5, Matrix Modifier - Citric Acid & Palladium
Standard 1 = 5 ppb, 2 = 10 ppb, 3 = 25 ppb, 4 = 50 ppb
ICV = 25 ppb, CCV = 25 ppb

QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within ± 20% of true value.

At least one preparatory blank, laboratory standard, spike and duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See below.)

*Method of standard additions:*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

1) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

3) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume $V_x$, are taken. To the first (labeled A) is added a

small volume $V_s$ of a standard analyte solution of concentrate $c_s$. To the second (labeled B) is added the same volume $V_s$ of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration $c_x$ is calculated:

$$c_x = \frac{S_B V_S c_S}{(S_A - S_B) V_X}$$

where,

$S_A$ and $S_B$ are the analytical signals (corrected for the blank) of solutions A and B, respectively. $V_S$ and $c_s$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_S$ is made much less than $V_X$, and thus $c_s$ is much greater than $c_x$, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

DATA TREATMENT:

For determination of metal concentration by direct aspiration and fumace; read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

$$\text{ug/L metal in sample} = A \times \frac{(C + B)}{C}$$

where,

A = ug/L of metal in diluted aliquot from calibration curve
B = Acid blank matrix used for dilution, mL
C = sample aliquot, mL

DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration Determined and resulting EQL
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- Spike Sample Recovery form
- Laboratory Control Sample Summary form
- All Raw Data
- Preparation Records

# STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF NON-AQUEOUS SAMPLES USING SONICATION BY SW-846 METHOD 3550B

Originating Author: Karin Stewart
Revision Author: Jeff Loewe

This SOP is effective upon signed approval by the following:

Unit Supervisor — Date: 6/26/01

QA/QC Director — Date: 6.20.2001

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

## 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This procedure is for the preparation of samples for Pesticide, Polychlorinated Biphenyl, Polyaromatic Hydrocarbon, and Semi-Volatile Organic Analytes using ultrasonic extraction. This procedure is applicable to the preparation of solid matrix samples.

## 3.0 SUMMARY

3.1 This process involves the isolation and concentration of organic compounds from non-aqueous samples for a variety of chromatographic techniques. The sonication process is used to ensure intimate contact of the sample matrix with the extraction solvent.

3.2 A measured volume of sample, usually 30 grams, is mixed with a drying agent to form a free flowing powder. This mixture is then serially extracted with solvent using a sonicator. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or analytical method used.

## 4.0 DEFINITIONS

4.1 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.2 Analyte – The specific component measured in a chemical analysis.

4.3 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.
- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Method Blank.

4.4 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.5 Laboratory Control Sample (LCS) – An aliquot of laboratory pure reagent spiked with target analytes or compounds representative of target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.6 Laboratory Control Sample Duplicate (LCSD) – An aliquot of laboratory pure reagent spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.7 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.8 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.9 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.10 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.11 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.12 Sample – A portion of material supplied by the client for analysis.

4.13 Sample Duplicate – Two aliquots of the same sample processed independently. This monitors precision of the analysis. Precision results are reported as relative percent difference (RPD).

4.14 Solvent exchange – Adding a different solvent other than the original extraction solvent and evaporating off the original solvent.

## 5.0 INTERFERENCES

5.1 Interferences that co-elute vary considerably from sample to sample.

5.2 If the analysis of an extracted sample is prevented due to matrix interferences, further clean-up of the extract may be required.

5.3 Phthalate esters can contaminate many types of plasticware and glassware products used in the lab. Plastics, in particular, must be avoided because phthalates are commonly used in plasticizers and are easily extracted from plastic materials. Phthalate contamination may easily result any time that consistent adherence to the quality control requirements are not practiced.

5.4 Soap residue may cause the degradation of certain analytes especially aldrin, heptachlor, and most organophosphorus pesticides. Strict adherence to the Glassware Washing SOP is required.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Turbo Vap II concentrator (water bath = 33°C) and tubes

7.3 Sonicator

7.4 250 ml glass beakers

7.5 Glass funnels

7.6 Filter paper (Fisher P-8 or equivalent)

7.7 Disposable pipettes, 1 and 10 ml

7.8 Graduated cylinders, glass, 100 and 1000 ml

7.9 2 ml autosampler vials and caps

7.10 Test tubes with caps

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook.

8.2 Reagents

8.2.1 Lab pure water

8.2.2 Acetone ($C_3H_6O$)

8.2.3 Acetonitrile ($C_2H_3N$)

8.2.4 Hexane ($C_6H_{14}$)

8.2.5 Methanol ($CH_4O$, also noted as MeOH)

8.2.6 Methylene chloride ($CH_2Cl_2$)

8.2.7 Sodium sulfate ($Na_2SO_4$)

8.3 Standards

8.3.1 Stock Base-Neutral Spike Standard, 1000 ug/ml each: Supelco #502294. See table in section 18.0 for compound list.

8.3.2 Stock Acid Spike Standard, 2000 ug/ml each: Supelco #502308. See table in section 18.0 for compound list.

8.3.3 SVOA Spike: In a 50 ml volumetric flask, dilute 2.5 ml of the stock base-neutral spike standard and 2.5 ml of the stock acid spike standard to the mark with MeOH. This prepares a standard containing the base-neutral compounds at 50 ug/ml and the acid compounds at 100 ug/ml. **Add 1 ml of this solution to the LCS, MS, and MSD samples.**

8.3.4 Stock Base-Neutral Surrogate Standard, 5000 ug/ml each: Supelco #4-7262. See table in section 18.0 for compound list.

8.3.5 Stock Acid Surrogate Standard, 10,000 ug/ml each: Supelco #4-7261. See table in section 18.0 for compound list.

8.3.6 SVOA Surrogate Standard: In a 100 ml volumetric flask, dilute 2.0 ml of the stock base-neutral surrogate standard and 1.5 ml of the stock acid surrogate standard to the mark with MeOH. This prepares a standard containing the base-neutral compounds at 100 ug/ml and the acid compounds at 150 ug/ml. **Add 1 ml of this solution to all samples.**

8.3.7 PNA-IL Surrogate Standard, 10 ug/ml each: Ina 200 ml volumetric flask, dilute 500 ul of the stock base-neutral surrogate standard to the mark with MeOH. **Add 1 ml of this solution to all samples.**

8.3.8 Stock PNA-IL Spike Standard, 2000 ug/ml each: Accustandard #Z-014G-R-PAK.

8.3.9 PNA-IL Spike Standard, 10 ug/ml each: In a 100 ml volumetric flask, dilute 500 ul of the stock PNA-IL spike standard to the mark with MeOH. **Add 1 ml of this solution to the LCS, MS, and MSD samples.**

8.3.10 Stock Phenol Surrogate Standard, 2000 ug/ml each: Accustandard #M-8040-SS-PAK contains 2-Fluorophenol and 2,4,6-Tribromophenol.

8.3.11 Phenol Surrogate Standard, 100 ug/ml: In a 25 ml volumetric flask, dilute 1.25 ml of the stock phenol surrogate standard to the mark with acetone. **Add 1 ml of this solution to all samples.**

8.3.12 Stock HPLC PNA Surrogate Standard, 2000 ug/ml: Accustandard #M-625-04-10X contains Decachlorobiphenyl (DCB).

8.3.13 HPLC PNA Surrogate Standard, 50 ug/ml: In a 50 ml volumetric flask, dilute 1.25 ml of the stock HPLC PNA surrogate standard to the mark with acetonitrile. **Add 1 ml of this solution to all samples.**

8.3.14 Stock PCB Spike Standard, 1000 ug/ml: Supelco #4-4809 contains Aroclor 1260.

8.3.15 PCB Spike Standard, 5 ug/ml: In a 100 ml volumetric flask, dilute 500 ul of the stock PCB spike standard to the mark with hexane. **Add 1 ml of this solution to the LCS, MS, and MSD.**

8.3.16 Stock Pesticide Spike Standard, 2000 ug/ml each: Supelco #4-8913. See the table in section 18.0 for the compound list.

8.3.17 Pesticide Spike Standard, 0.5 ug/ml each: In a 100 ml volumetric flask, dilute 25 ul of the stock pesticide spike standard to the mark with acetone. **Add 1 ml of this solution to the LCS, MS, and MSD.**

8.3.18 Stock Pest/PCB Surrogate Standard, 200 ug/ml each: Accustandard #CLP-032-K contains DCB and TCMX.

8.3.19 Pest/PCB Surrogate Standard, 0.2 ug/ml each: In a 200 ml volumetric flask, dilute 200 ul of the stock pest/PCB surrogate standard to the mark with hexane. **Add 1 ml of this solution to all samples.**

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Samples should be collected in a clean glass container. Preservation consists of storage in the range of 0.1-6°C.

9.3 Preparation must be performed within the maximum allowable hold time of 14 days from collection.

## 10.0 QUALITY CONTROL

10.1 A *Laboratory Control Standard* must be extracted with each batch of maximum 20 samples and at a minimum of one per day analyzed.

10.2 A *Method Blank* must be extracted with each LCS.

10.3 A *Matrix Spike and Matrix Spike Duplicate* sample must be extracted with each group of maximum 10 samples at a minimum of one per day extracted for the 600 series methods with the exception of method 625, and with each group of maximum 20 samples at a minimum of one per day extracted for the 8000 series methods and method 625. If insufficient sample exists for the preparation of a MS/MSD, a duplicate LCS (i.e. LCSD) should be extracted.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Perform the required preventative maintenance as necessary.

11.2 Check water level in the Turbo Vap II. Add DI water as needed.

11.3 Check temperature of the Turbo Vap II water bath and adjust as needed.

11.4 Verify or calibrate the balance on a daily basis.

## 12.0 PROCEDURE

12.1 Rinse all glassware and the sonicator probe with acetone.

12.2 Triple rinse all glassware and the sonicator probe with methylene chloride.

12.3 Transfer 30 g of a well mixed sample into beaker and record weight to the nearest tenth of a gram.

12.4 Add enough sodium sulfate to beaker to dry sample, and mix.

12.5 Add surrogates and spikes to appropriate samples and record type, lot number, and amount added.

12.6 Add 60 ml methylene chloride to beaker.

12.7 Sonicate for 3 minutes.

12.8 Prepare a funnel with filter paper and sodium sulfate.

12.9 Rinse filter with approximately 20 ml of methylene chloride and discard the methylene chloride.

12.10 Pour extract from beaker through filter into a concentrator tube.

12.11 Repeat steps 12.6, 12.7,and 12.10 two more times.

12.12 Rinse filter with methylene chloride adding this to the concentrator tube.

12.13 Place concentrator tube into Turbo Vap II.

12.14 If needed, "solvent exchange" the extract when its volume is below 1 ml and continue to evaporate extract to below 1 ml then remove from the Turbo Vap II. If extract does not need a solvent exchange, simply remove extract from Turbo Vap II when extract falls below 1 ml. **CAUTION: DO NOT LET EXTRACT GO DRY!** See table in section 18.0 for the required final solvent.

12.15 Using a 1 ml syringe, measure and remove extract from concentrator tube and place in appropriate container (e.g. vials, test tubes, etc.) labeled with the sample I.D., fraction, volume, parameter, and extraction personnel initials.

12.16 Rinse concentrator tube walls with 2-3 ml of appropriate solvent.

12.17 Using rinse solvent in concentrator tube, adjust volume of extract appropriate volume.

12.18 Cap container of extract.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 Enter sample preparation data into the LIMS system.

## 14.0 METHOD PERFORMANCE

14.1 Not applicable.

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Dispose of any resulting residue, digestate, or extract in accordance with local sanitary regulations.

16.2 Additional sample shall be disposed of properly following the completion of analysis and an appropriate additional holding time.

## 17.0 REFERENCES

17.1 SW-846 Method 3550B

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

| BN Spike Standard Compounds | |
|---|---|
| Acenapthene | 1,2,4-Trichlorobenzene |
| N-Nitrosodi-N-propylamine | 1,3-Dichlorobenzene |
| Pyrene | 2,4-Dinitrotoluene |

| Acid Spike Standard Compounds | |
|---|---|
| Pentachlorophenol | 4-Chloro-3-methylphenol |
| Phenol | 4-Nitrophenol |
| 2-Chlorophenol | |

| BN Surrogate Standard Compounds | |
|---|---|
| Nitrobenzene-d5 | 1,2-Dichlorobenzene-d4 |
| p-Terphenyl-d14 | 2-Fluorobiphenyl |

| Acid Surrogate Standard Compounds | |
|---|---|
| Phenol-d6 | 2,4,6-Tribromophenol |
| 2-Chlorophenol-d4 | 2-Fluorophenol |

| PNA Spike Standard Compounds | |
|---|---|
| Naphthalene | Benzo(b)fluoranthene |
| Acenaphthene | Benzo(k)fluoranthene |
| Acenaphthylene | Benzo(a)pyrene |
| Flourene | Dibenzo(a,h)anthracene |
| Pyrene | Benzo(g,h,i)perylene |
| Benzo(a)anthracene | Indeno(1,2,3-cd)pyrene |
| Chrysene | |

| Pesticide Spike Standard Compounds | |
|---|---|
| Aldrin | Endrin aldehyde |
| Alpha-BHC | Endrin ketone |
| Beta-BHC | Gamma-BHC |
| Delta-BHC | Heptachlor |
| Dieldrin | Heptachlor epoxide |
| Endosulfan I | Methoxychlor |
| Endosulfan II | 4,4'-DDD |
| Endosulfan sulfate | 4,4'-DDE |
| Endrin | 4,4'-DDT |

| Extraction Conditions | | | | | |
|---|---|---|---|---|---|
| Determinative Method and Prep Code | Analyte Group | Initial Extraction pH | Final Solvent for Analysis | Final Solvent for Cleanup | Final Vol., ml |
| 8041<br>3510_Phenol | Phenols | ≤ 2 | Hexane | --- | 1 |
| 8081A<br>3510_Pest | Pesticides | 5 – 9 | Hexane | Hexane | 10 |
| 8082<br>3510_PCB | PCBs | 5 – 9 | Hexane | Hexane | 10 |
| 8270C[1]<br>3510_B | SVOA (BNA) | <2 | None | --- | 1 |
| 8310<br>3510_HPLC | PAH (PNA) | As received | Acetonitrile | --- | 1 |

1 = Extraction pH sequence may be reversed to better separate the acid and neutral components. Excessive pH adjustments may result in the loss of some analytes.

# STANDARD OPERATING PROCEDURE FOR POLYNUCLEAR AROMATIC HYDROCARBONS BY EPA METHOD 610 AND SW-846 METHOD 8310

Originating Author: Unknown
Revision Author: Christine Robinson

This SOP is effective upon signed approval by the following:

Unit Supervisor — Date: 12/21/01

QA/QC Director — Date: 12-21-2001

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

## 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This is a High Performance Liquid Chromatography (HPLC) procedure for the determination of Polynuclear Aromatic Hydrocarbons (PNA or PAHs). This procedure is applicable to the analysis of extracts from aqueous, non-aqueous liquid, and solid matrix samples. The routine reporting limits are listed in the table below. These limits may vary due to sample matrix.

| ANALYTE | PQL, ug/l | PQL, ug/kg |
|---|---|---|
| Acenaphthene | 5 | 800 |
| Acenaphthylene | 2.5 | 400 |
| Anthracene | 0.1 | 16 |
| Benzo(a)anthracene | 0.1 | 16 |
| Benzo(a)pyrene | 0.2 | 32 |
| Benzo(b)fluoranthene (3,4-Benzofluoranthene) | 0.1 | 16 |
| Benzo(g,h,i)perylene | 0.4 | 64 |
| Benzo(k)fluoranthene | 0.1 | 16 |
| Chrysene | 0.2 | 32 |
| Dibenz(a,h)anthracene | 0.3 | 48 |
| Fluoranthene | 0.25 | 40 |
| Fluorene | 0.5 | 80 |
| Indeno(1,2,3-cd)pyrene | 0.25 | 40 |
| Naphthalene | 2.5 | 400 |
| Phenanthrene | 0.2 | 32 |
| Pyrene | 0.5 | 80 |

## 3.0 SUMMARY

3.1 PAHs are extracted from the sample matrix using methylene chloride. Extracts are concentrated and exchanged into acetonitrile prior to analysis. The extract is injected into a HPLC having an Ultraviolet (UV) and a Fluorescence detector as specified in Methods 610 and 8310.

3.2 The linear working range varies depending on the compound. See the table of calibration standard concentration for compound specific ranges.

## 4.0 DEFINITIONS

4.1 Accuracy – The degree of agreement of a measured value with the true or expected value of the quantity of concern (% recovery of a known spiked analyte).

4.2 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.3 Analyte – The specific component measured in a chemical analysis.

4.4 Analytical Batch – A group of samples which are analyzed, at the instrument level, together using the same method, reagents and apparatus within the same time period. Typically, these are samples in the same batch ID in the LIMS.

4.5 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Calibration Blank – An aliquot of the standard diluent (water or organic solvent) that is not carried through the sample preparation scheme. It is analyzed to verify that the analytical system is free from contamination. Also referred to as an instrument blank or solvent blank.

- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.

- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Method Blank.

4.6 Bias – The deviation of a measured value from a known or accepted value due to matrix effects or method performance. Bias may be determined quantitatively to correct measured values. Bias may be positive or negative.

4.7 Breakdown – A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

4.8 Calibration – The establishment of an analytical curve based on the absorbance, response, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type and concentration of acids, solvents, or other solutions used in the sample preparation.

4.9 Calibration Factor (CF) – A measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the volatile and semi-volatile fractions.

$$CF = \frac{\text{Area of the compound in the standard}}{\text{Mass of the compound (in ng units)}}$$

4.10 Confirmation – In gas chromatography, an unknown compound in a sample is identified based upon its retention time on a specific chromatographic column. Because several compounds may exhibit the exact same retention time on a given column, a secondary analysis on a different column or detector is often required for additional confidence in the compound identification.

4.11 Continuing Calibration Verification Standard (CCV) – A standard used to verify the continued acceptability of the initial calibration curve. A continuing calibration

verification must be repeated at the beginning and end of each analytical batch and every 10-20 samples, whichever is more frequent depending on the method requirements. The concentrations of the continuing calibration verification standard shall be varied within the established calibration range. If an internal standard is used, only one continuing calibration verification must be analyzed per analytical batch.

4.12 Detection Limit – The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

- MDL – Method detection limit. The minimum concentration of a substance that can be measured and reported with a 99% degree of confidence. MDLs are determined by analyzing a minimum of seven consecutive standards that have been processed through all preparatory steps.

- PQL – The Practical Quantitation Limit is the lowest concentration that can reliably be achieved within specified limits of precision and accuracy during routine laboratory operating conditions. Typically, the PQL is a value in the range of 5 - 10 times the MDL. This is the reporting limit and is also referred to as the Estimated Quantitation Limit (EQL).

4.13 Initial Calibration Verification (ICV) – A standard used to verify the accuracy of calibration standards. Prepared from a second source than that of the calibration standards, its known value is measured against the calibration curve. This determines the integrity of working standards. Also referred to as an external verification standard or check standard.

4.14 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.15 Internal Standard (ISTD) – Applicable to GC/MS and LC analyses only. A compound(s), not of interest as a target compound, which is added to all samples, QC samples, and calibration standards just prior to instrument analysis. Internal standards are used as the basis for quantitation of target compounds for GC/MS analysis.

4.16 Laboratory Control Sample (LCS) – An aliquot of laboratory pure reagent spiked with target analytes or compounds representative of target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.17 Laboratory Control Sample Duplicate (LCSD) – An aliquot of laboratory pure reagent spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.18 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or

other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.19 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.20 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.21 Mean Calibration Factor – The average of the calibration factors obtained through the initial calibration.

$$CF_{mean} = (CF_1 + CF_2 + CF_3 + \ldots CF_n) / n$$

Where, $CF_1$ is the CF from standard 1, $CF_2$ is the CF from standard 2, etc.
n is the total number of calibration standards

4.22 Percent Difference (%D) – Used to compare two values, the percent difference indicates both the direction and the magnitude of the comparison. The percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

$$\%D = \frac{(X - Y)}{X} * 100$$

where: X = value 1
Y = value 2

4.23 Percent Recovery – A measure of accuracy that is calculated as the measured value relative to the true value, expressed as a percent.

$$\%R = \frac{MV}{TV} * 100$$

where: MV = measured value
TV = true value

4.24 Precision – The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the comparability of results from duplicate or

replicate analyses. (%RPD between the recoveries of two known analyte spikes, and %RSD between the recoveries of three or more measurements).

4.25 Preparation Batch -- A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent. Typically, these are samples in the same batch ID in the LIMS.

4.26 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.27 Relative Percent Difference (% RPD) – Used to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

$$\% \text{ RPD} = \frac{|X - Y|}{(X + Y) / 2} * 100$$

where: X = value 1
Y = value 2

4.28 Relative Retention Time (RRT) – The ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$\text{RRT} = \frac{RT_c}{RT_{is}}$$

where: $RT_c$ = Retention time for the target or surrogate in continuing calibration
$RT_{is}$ = Retention time for the internal standard in calibration standard or sample

4.29 Relative Standard Deviation – Statistical parameter used to measure the variability of a data set with respect to the mean of that data set.

$$\%\text{RSD} = (\text{SD} / \text{mean}) * 100$$

4.30 Retention Time – The time elapsed from sample injection until the specific compound elutes or exits the chromatographic column at the detector. Each analyte has a characteristic retention time on a specific column allowing this information is used to qualitatively identify the analytes in the sample.

4.31 Sample – A portion of material supplied by the client for analysis.

4.32 Standard Deviation (SD) - A statistical parameter that indicates the variability of a data set as centered on the mean.

$$SD = \frac{\sqrt{\sum(x_i - x_{mean})^2}}{n-1}$$

where, $x_i$ is an individual value
$x_{mean}$ is the average of all values
n is the total number of values in the data set

4.33 Surrogate Compound – Compound that behaves similarly, with respect to the analytical method, as the analytes of interest but is not normally found in environmental samples. Often, surrogates are isotopic homologues of target analytes. Surrogate(s) are added to all blanks, samples and QC samples prior to preparation and analysis. Recovery of surrogates is used to assess method performance.

## 5.0 INTERFERENCES

5.1 Contamination by carryover can occur when a low-level sample is analyzed after a high-level sample. Solvent blanks should be analyzed in these instances to check for effects from carryover.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Hewlett Packard 1050 HPLC with UV and Fluorescence detectors

7.3 Chromatographic column: Supelco LC-PAH. Dimensions are 15cm X 4.6mm, 5 um particle size.

7.4 Computer with Chemstation software, monitor and printer.

7.5 Syringes: Various sizes including 10, 500, and 1000 ul.

7.6 Autosampler vials: 2 ml size with screw tops.

7.7 200 ul glass inserts.

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

8.2.1 Analyte free water (HPLC Grade) is purchased and has been filtered through a 0.1-micron filter.

8.2.2 Acetonitrile ($C_2H_3N$) – HPLC quality, distilled in glass

8.3 Standards

8.3.1 Stock HPLC PNA Surrogate Standard, 2000 ug/ml: Accustandard catalog #M-625-04 contains Decafluorobiphenyl (DFB) in methylene chloride. Store in the Organics standard freezer.

8.3.2 Stock Calibration Standard: Ultra catalog #PM-831A (or equivalent) contains the compounds in a mix of acetonitrile and methanol. See the table in section 18.0 for the compound specific concentrations. Transfer the standard to a glass container having a Teflon lined cap. Store the standard in the Organics standard freezer and replace after one year, or sooner if necessary.

8.3.3 Working Linearity Curve: In separate 1 ml volumetric flasks, prepare the dilutions listed in section 18.0.

8.3.4 Stock Verification (ICV) Standard: Supelco catalog #4-9156 (or equivalent). The concentrations are the same as those in the stock calibration standard (see section 18.0). Transfer the standard to a glass container having a Teflon lined cap. Store the standard in the Organics standard freezer and replace after one year, or sooner if necessary.

8.3.5 Working ICV: In a 1.0 ml volumetric flask, dilute 50 ul of the stock verification standard to the mark with acetonitrile. This prepares a standard of concentrations equal to the PAH level 3 linearity standard (see section 18.0).

8.3.6 LCS/MS/MSD: When prepared as detailed in the sample preparation SOP, the LCS/MS/MSD will contain the following analytes.

| COMPOUND | CONC., ug/l |
|---|---|
| Anthracene<br>Benzo(b)fluoranthene<br>Benzo(k)fluoranthene | 0.8 |
| Phenanthrene | 1.6 |
| Benzo(a)anthracene<br>Benzo(a)pyrene<br>Chrysene<br>Fluoranthene<br>Indeno(1,2,3-cd)pyrene | 2.0 |
| Benzo(g,h,i)perylene | 3.2 |
| Fluorene<br>Pyrene | 4.0 |
| Dibenz(a,h)anthracene | 8.0 |
| Acenaphthylene<br>Naphthalene | 20.0 |
| Acenaphthene | 40.0 |

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Extracts are stored at -15°C in the extract freezer located in the SVOA lab.

9.3 Analysis must be performed within the maximum allowable hold time of 40 days from extraction.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Analyze four separate standards prepared in the range of 8-10 times the method detection limit listed in section 14.0. These standards must be from a source different from that used for calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.2 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Analyze a minimum of seven (maximum of ten) standards prepared in the range of 2-5 times the method detection limit listed in section 14.0 or an estimated detection limit. These standards must be taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.3 *Surrogate* (SURR) standard must be added to all quality control samples, blanks, and samples. Acceptance criteria are the statistically generated recovery limits of 13.8 – 106% recovery. These criteria are evaluated by the LIMS and printed on the LIMS QC Report and analytical report submitted to the client. Failures are automatically flagged with a "S" on these reports. If the acceptance criteria are not met, re-extract and analyze. If reanalysis does not yield acceptable recovery, both sample sets must be supplied to the client. If reanalysis is performed beyond the maximum allowable hold time, both sample sets must be supplied to the client and the appropriate result flagged with a "H" qualifier as defined in the LIMS. If insufficient sample is available for re-extraction the original result should be reported and qualified in the Case Narrative to state this as the reason for no re-extraction and reanalysis being performed. If the acceptance criteria are not met for a method blank but are met for the QC samples and environmental samples, report the sample results qualified for the MB failure in the Case Narrative.

10.4 An *Initial Calibration Verification* (ICV) standard must be immediately after the initial linearity (ICAL). This is the analysis of a second source standard. Acceptance criteria are the statistically generated recovery limits below. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, analysis must be stopped, the problem corrected, and the instrument recalibrated. The LIMS will evaluate the recovery criteria and flag not acceptable recoveries with a "S" flag.

| Analyte | Low | High |
|---|---|---|
| Acenaphthene | 84.9 | 108 |
| Acenaphthylene | 86.3 | 115 |
| Anthracene | 84.2 | 112 |
| Benzo[a]anthracene | 87.1 | 122 |
| Benzo[a]pyrene | 91.2 | 122 |
| Benzo[b]fluoranthene (3,4-Benzofluoranthene) | 85.4 | 119 |
| Benzo[g,h,i]perylene | 79.4 | 114 |
| Benzo[k]fluoranthene | 79.4 | 123 |
| Chrysene | 88 | 118 |
| Dibenz[a,h]anthracene | 67 | 127 |
| Fluoranthene | 82.9 | 112 |
| Fluorene | 88.8 | 108 |
| Indeno[1,2,3cd]pyrene | 86.6 | 112 |
| Naphthalene | 88.7 | 119 |
| Phenanthrene | 83.3 | 109 |
| Pyrene | 88.6 | 116 |

10.5 A *Continuing Calibration Verification* (CCV) standard is a calibration source standard. The initial calibration must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. The 12-hour shift begins with the injection of the CCV1 standard, continues through the analysis of environmental samples (maximum of 20), and ends with the injection of the CCV2 standard (which must be injected within 12-hours of the start of the sequence). The concentration of the CCV must be varied throughout the run (see the calibration standards for the concentrations of CCV1 and CCV2). Acceptance criteria are $\leq$ 15% difference when compared to the mean calibration factor of the initial calibration. In keeping with the approach of averaging as in the Calibration

section, if the response for a given analyte is > 15% difference but the average of all analytes is ≤ 15% difference, the calibration is considered verified and acceptable. If acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, recalibrate. When a CCV fails to meet the acceptance criteria, recalibrate. When a CCV fails to meet the acceptance criteria, all samples that are not bracketed by acceptable verifications must be re-injected. Samples with a non-detect concentration may be reported if the CCV fails to meet the acceptance criteria with a positive bias. Initial and date the Evaluate Continuing Calibration Report and the Quantitation Report to indicate that the acceptance criteria have been reviewed.

10.6 A *Method Blank* (MB) must be extracted and analyzed with each batch of up to 10 samples per day for Method 610 and 20 samples per day for Method 8310. Acceptance criteria are no detects above the PQL, however, the MB remains acceptable if the blank concentration is less than 1/10 of the sample concentration, or if there were no detects in the sample. If the acceptance criteria are not met, re-extract the blank and the affected samples if sufficient sample is available. If insufficient sample is available for reanalysis the original result should be reported and qualified in the Case Narrative. Samples associated with a contaminated blank must be reported with a "B" qualifier as defined in the LIMS. If sample reanalysis was performed beyond the maximum allowable hold time, both sets of results must be supplied to the client and flagged with a "H", as appropriate, to note the hold time exceedance. When a MB is contaminated, corrective action steps must be taken to identify and eliminate the cause of the contamination.

10.7 A *Laboratory Control Sample* (LCS) must be extracted and analyzed with each batch of up to 10 samples per day for Method 610 and 20 samples per day for Method 8310, per matrix. Acceptance criteria (listed below) are the statistically generated recovery limits for Method 8310 or the recovery limits in Table 3 of Method 610, as appropriate. However, the LCS remains acceptable if the failed recovery is positive bias (high) and there are no detects in the sample. If the acceptance criteria are not met, reanalyze or re-extract as appropriate. If this reanalysis does not meet the acceptance criteria, the affected samples from that batch must be re-extracted and analyzed. If insufficient sample is available for reanalysis the original result should be reported and qualified in the Case Narrative. If the hold time has expired and reanalysis performed, both sets of data should be reported and the appropriate result flagged with a "H" qualifier as defined in the LIMS.

| LCS Recovery Limits | | | | |
|---|---|---|---|---|
| | 8310 | | 610 | |
| Analyte | Low | High | Low | High |
| Acenaphthene | 35.5 | 150 | 5 | 124 |
| Acenaphthylene | 32.7 | 140 | 5 | 139 |
| Anthracene | 29.1 | 155 | 5 | 126 |
| Benzo[a]anthracene | 42.8 | 157 | 12 | 135 |
| Benzo[a]pyrene | 19.5 | 193 | 5 | 128 |
| Benzo[b]fluoranthene | 7.01 | 166 | 6 | 150 |
| Benzo[g,h,i]perylene | 9.49 | 127 | 5 | 116 |
| Benzo[k]fluoranthene | 5 | 152 | 5 | 159 |
| Chrysene | 35.4 | 155 | 5 | 199 |

| | | | | |
|---|---|---|---|---|
| Dibenz[a,h]anthracene | 5 | 123 | 5 | 110 |
| Fluoranthene | 30.2 | 171 | 14 | 123 |
| Fluorene | 29 | 156 | 5 | 142 |
| Indeno[1,2,3cd]pyrene | 10.5 | 141 | 5 | 116 |
| Naphthalene | 25.2 | 139 | 5 | 122 |
| Phenanthrene | 32.7 | 157 | 5 | 155 |
| Pyrene | 41.9 | 163 | 5 | 140 |

10.8 A *Matrix Spike and Matrix Spike Duplicate* (MS/MSD) must be extracted and analyzed with each batch of up to 10 samples per day for Method 610 and 20 samples per day for Method 8310. When insufficient sample is available for a MSD, a duplicate laboratory control sample (LCSD) should be extracted and analyzed. Acceptance criteria are the statistically generated recovery limits for Method 8310 or the recovery limits in Table 3 of Method 610, as appropriate, however, the MS remains acceptable if the failed recovery is positive bias (high) and there are no detects in the sample. If the acceptance criteria are not met for the MS, evaluate the MSD for accuracy. If the acceptance criteria are met in the MSD, continue. If the accuracy criteria are not met in the MS or MSD, and the LCS is in control, assume matrix interference and report the results with a "S" qualifier as defined in the LIMS. Precision criteria are the statistically generated limits listed in section 18.0. If the precision criteria are not met, report the results wit a "R" qualifier as defined in the LIMS.

| MS/MSD Recovery Limits | | | | | | |
|---|---|---|---|---|---|---|
| | 8310 | | | 610 | | |
| Analyte | Low | High | RPD | Low | High | RPD |
| Acenaphthene | 35.5 | 150 | 36.8 | 5 | 124 | 36.8 |
| Acenaphthylene | 32.7 | 140 | 64.1 | 5 | 139 | 64.1 |
| Anthracene | 29.1 | 155 | 29.8 | 5 | 126 | 29.8 |
| Benzo[a]anthracene | 42.8 | 157 | 33.6 | 12 | 135 | 33.6 |
| Benzo[a]pyrene | 19.5 | 193 | 37.3 | 5 | 128 | 37.3 |
| Benzo[b]fluoranthene (3,4-Benzofluoranthene) | 7.01 | 166 | 24.2 | 6 | 150 | 24.2 |
| Benzo[g,h,i]perylene | 9.49 | 127 | 35.8 | 5 | 116 | 35.8 |
| Benzo[k]fluoranthene | 5 | 152 | 35.1 | 5 | 159 | 35.1 |
| Chrysene | 35.4 | 155 | 26.8 | 5 | 199 | 26.8 |
| Dibenz[a,h]anthracene | 5 | 123 | 49 | 5 | 110 | 49 |
| Fluoranthene | 30.2 | 171 | 31.1 | 14 | 123 | 31.1 |
| Fluorene | 29 | 156 | 46 | 5 | 142 | 46 |
| Indeno[1,2,3cd]pyrene | 10.5 | 141 | 37.2 | 5 | 116 | 37.2 |
| Naphthalene | 25.2 | 139 | 63.7 | 5 | 122 | 63.7 |
| Phenanthrene | 32.7 | 157 | 34.9 | 5 | 155 | 34.9 |
| Pyrene | 41.9 | 163 | 31.2 | 5 | 140 | 31.2 |

10.9 *Confirmation* of sample detects are made by detection on the alternate detector. While all the potential target PNA compounds (listed in section 2.1) are detectable by the UV detector, all but Acenaphthene, Acenaphthylene, Fluorene, Indeno(1,2,3-cd)pyrene, and Naphthalene are detectable with the fluorescence detector. As a result, detects for all but the above mentioned analytes are confirmed through the routine use of the alternate detector. Confirmations are performed to verify the identification of a target compound and not the concentration. In order to be used for confirmation, it must be demonstrated that

the other detector is sensitive at or below the level indicated in the initial analysis. This demonstration is accomplished through the acceptable analysis of the CCV1. Alternatively, if the initial linearity criteria were not met on the alternate detector, a standard of that particular analyte must be analyzed at a level at or below the concentration measured in the initial analysis. Identification of that analyte demonstrates sensitivity for that analyte at that level. Confirmation of those analytes not detectable on the alternate detector must be performed if requested by the client. If requested, confirmation analysis must be performed by GC/MS SIM or through the use of another column in the HPLC.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Perform the required preventative maintenance as necessary. A new Initial Calibration (linearity) is required as indicated by the quality control elements. The concentration of the low standard must be greater than the MDL and set near or below the routine PQL for each compound.

11.2 Instrument conditions are as follows:

| PAHTEST.m | |
|---|---|
| Flow rate: | 1.200 ml/min |
| Solvent A: | Water |
| Solvent B: | Acetonitrile (ACN) |
| Solvent mix: | 50% ACN at 0.00 minutes |
| | 50% ACN at 5.00 minutes |
| | 75% ACN at 15.00 minutes |
| | 100 % ACN at 30.00 minutes |
| | 100% ACN at 40.00 minutes |
| Injection size: | 30 ul |

| Fluorescence Detector | |
|---|---|
| Excitation: | 254 nm |
| Emission: | 420 nm |
| Pmtgain: | 10 |
| Response time: | 2.0 seconds |

| UV Detector | |
|---|---|
| Wavelength: | 254 nm |
| Response time: | 4.0 seconds |
| Peak width: | 0.532 minutes |

11.3 Set up sequence parameters in *Sequence* in main menu.

11.4 Set up analysis sequence in Sample Table in *Sequence* in main menu.

11.5 Enter the sequence as it is to be run, including the applicable analysis method. An ICV standard must be analyzed following the generation of a new linearity and the acceptance criteria met before continuing with sample analysis. If the acceptance

criteria are met, rename the method "xxxxxxx", where mmdd designates the month and date of the new linearity.

11.6 Transfer approximately 1 ml of extract to a 2 ml autosampler vial and cap the vial.

11.7 Load the autosampler tray.

11.8 Click on *Run Sequence* in Sample Table.

11.9 After each sample has run, the report is retrieved and quantitated against the current initial linearity. The chromatogram is examined to determine that all compounds present have been detected. The Quantitation Report indicates that quantitation is measured against the Initial Calibration.

11.10 A minimum 5 level calibration is performed. The mean CF must be < 10% RSD for Method 610, and < 20% RSD for Method 8310. Typically, the UV detector is used as the "primary" detector. The Fluorescence detector may be used as needed, based on calibration criteria, chromatographic interferences, etc. If the Fluorescence detector is used as the primary detector, it must be used as such for the calibration and all samples, and this fact must be documented on all instrument printouts. Where the calibration criteria are not met, the following options exist.

11.10.1 The mean of the RSD values for all analytes in the calibration is less than or equal to 20% for Method 8310 or 10% for Method 610, as appropriate. The mean RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. If no analyte has an RSD above 20% or 10% criteria, then the mean RSD calculation need not be performed.

11.10.2 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project. In other words, if the target analyte is part of the calibration standard, its RSD value is included in the evaluation.

11.11 If the linearity requirements are not met, take appropriate corrective actions and recalibrate. Analysis of environmental samples cannot proceed without the generation of an acceptable linearity.

## 12.0 PROCEDURE

12.1 Once it has been determined that the calibration and calibration verification standards meet their respective criteria, the analysis of samples can begin. A typical sequence follows the order: CCV1, MB, LCS, samples, QC samples, CCV2.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 After review, enter final results into the LIMS system. Results flagged by the LIMS with an "E" qualifier are above the linear range of the instrument. There is less

certainty in these data and, if sufficient sample and holding time are available, should be reanalyzed at an appropriate dilution. Details on the procedure for entering analytical data are in the Data Entry SOP. The peak integrations must be performed according to the Manual Integration of Chromatographic Peaks SOP.

13.2 Sample concentration is calculated as follows:

$$\text{Final Conc., ug/ml} = \frac{(A_{Sample})\,(V_f)\,(C_{Std})\,(DF)}{(A_{Std})\,(V_o)}$$

where: $A_{Sample}$ = Area of the sample
$V_f$ = Final volume, ml
$C_{Std}$ = Conc. of standard, ug/ml
DF = dilution factor
$A_{Std}$ = Area of the standard
$V_o$ = Initial sample size, L or kg

13.3 The LIMS calculates the dry-weight concentration for solid samples as follows:

$$\text{Conc. Dry} = \frac{\text{(wet weight conc.)}}{\text{(100 - \% Moisture)}}$$

## 14.0 METHOD PERFORMANCE

14.1 Method Detection Limit

The latest MDL study yielded the following data:

| Method Detection Limit Study | | | |
|---|---|---|---|
| Analyte | Spiked Conc., ug/l | Std. Dev. | MDL ug/l |
| Naphthalene | 1.25 | 0.1947 | 0.5842 |
| Acenaphthylene | 1.25 | 0.2536 | 0.7607 |
| Acenaphthene | 2.5 | 0.3564 | 1.0692 |
| Fluorene | 0.25 | 0.1027 | 0.3080 |
| Phenanthrene | 0.1 | 0.0759 | 0.2277 |
| Anthracene | 0.05 | 0.0345 | 0.1034 |
| Fluoranthene | 0.125 | 0.0506 | 0.1517 |
| Pyrene | 0.25 | 0.1343 | 0.4028 |
| Benzo(a)anthracene | 0.125 | 0.0295 | 0.0885 |
| Chrysene | 0.125 | 0.0138 | 0.0414 |
| Benzo(b)fluoranthene | 0.05 | 0.0186 | 0.0557 |
| Benzo(k)fluoranthene | 0.05 | 0.0406 | 0.1219 |
| Benzo(a)pyrene | 0.125 | 0.0515 | 0.1546 |
| Indeno(1,2,3-cd)pyrene | 0.125 | 0.0147 | 0.0442 |
| Dibenz(a,h)anthracene | 0.5 | 0.3657 | 1.0972 |
| Benzo(g,h,i)perylene | 0.2 | 0.0870 | 0.2610 |

14.2 Initial Demonstration of Capability

The latest IDC study yielded the following data:

| Initial Demonstration of Capability Study (n=4) | | | | |
|---|---|---|---|---|
| Analyte | Spiked Conc., ug/l | Avg. Conc., ug/l | Avg. %R | Std. Dev. |
| Naphthalene | 20 | 15.5 | 77.7 | 0.486 |
| Acenaphthylene | 20 | 16.2 | 80.7 | 0.552 |
| Acenaphthene | 40 | 31.8 | 79.6 | 1.13 |
| Fluorene | 4 | 3.35 | 83.8 | 0.123 |
| Phenanthrene | 1.6 | 1.28 | 80.2 | 0.059 |
| Anthracene | 0.8 | 0.68 | 85.0 | 0.024 |
| Fluoranthene | 2 | 1.56 | 77.8 | 0.056 |
| Pyrene | 4 | 3.53 | 88.3 | 0.158 |
| Benzo(a)anthracene | 2 | 1.78 | 88.8 | 0.080 |
| Chrysene | 2 | 1.72 | 85.8 | 0.074 |
| Benzo(b)fluoranthene | 0.8 | 0.93 | 91.6 | 0.044 |
| Benzo(k)fluoranthene | 0.8 | 0.68 | 84.7 | 0.039 |
| Benzo(a)pyrene | 2 | 1.70 | 84.9 | 0.064 |
| Indeno(1,2,3-cd)pyrene | 2 | 1.75 | 87.6 | 0.074 |
| Dibenz(a,h)anthracene | 8 | 6.87 | 85.8 | 0.302 |
| Benzo(g,h,i)perylene | 3.2 | 2.68 | 83.6 | 0.079 |

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Refer to the SIMALABS International Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, extract or standard.

## 17.0 REFERENCES

17.1 USEPA Method 610

17.2 SW-846 Method 8310

17.3 SIMALABS International SOP Preparation of Aqueous Samples Using Liquid-Liquid Extraction by SW-846 Method 3510C, current revision.

17.4 SIMALABS International SOP Preparation of Aqueous Samples Using Continuous Liquid-Liquid Extraction by SW-846 Method 3520C, current revision.

17.5 SIMALABS International SOP Preparation of Non-Aqueous Samples Using Sonication by SW-846 Method 3550B, current revision.

17.6 SIMALABS International Quality Assurance Plan, current revision

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

| STOCK CALIBRATION STANDARD (ULTRA #PM-831A) | |
|---|---|
| ANALYTE | ug/ml |
| Acenaphthene | 1000 |
| Acenaphthylene | 500 |
| Anthracene | 20 |
| Benzo(a)anthracene | 50 |
| Benzo(b)fluoranthene | 20 |
| Benzo(k)fluoranthene | 20 |
| Benzo(g,h,i)perylene | 80 |
| Benzo(a)pyrene | 50 |
| Chrysene | 50 |
| Dibenz(a,h)anthracene | 200 |
| Fluoranthene | 50 |
| Fluorene | 100 |
| Indeno(1,2,3-cd)pyrene | 50 |
| Naphthalene | 500 |
| Phenanthrene | 40 |
| Pyrene | 100 |

| PAH LEVEL 5 LINEARITY STANDARD (200 ul of stock calibration standard plus 200 ul stock surrogate to 1ml final volume) | |
|---|---|
| ANALYTE | ug/ml |
| Acenaphthene | 200 |
| Acenaphthylene | 100 |
| Anthracene | 4.0 |
| Benzo(a)anthracene | 10 |
| Benzo(b)fluoranthene | 4.0 |
| Benzo(k)fluoranthene | 4.0 |
| Benzo(g,h,i)perylene | 16 |
| Benzo(a)pyrene | 10 |
| Chrysene | 10 |
| Dibenz(a,h)anthracene | 40 |
| Fluoranthene | 10 |
| Fluorene | 20 |
| Indeno(1,2,3-cd)pyrene | 10 |
| Naphthalene | 100 |
| Phenanthrene | 8.0 |
| Pyrene | 20 |
| DFB (Surr) | 400 |

| PAH LEVEL 4 LINEARITY STANDARD* (1 ml of stock calibration standard plus 1 ml stock surrogate to 10 ml final volume) | |
|---|---|
| ANALYTE | ug/ml |
| Acenaphthene | 100 |
| Acenaphthylene | 50 |
| Anthracene | 2.0 |
| Benzo(a)anthracene | 5.0 |
| Benzo(b)fluoranthene | 2.0 |
| Benzo(k)fluoranthene | 2.0 |
| Benzo(g,h,i)perylene | 8.0 |
| Benzo(a)pyrene | 5.0 |
| Chrysene | 5.0 |
| Dibenz(a,h)anthracene | 20 |
| Fluoranthene | 5.0 |
| Fluorene | 10 |
| Indeno(1,2,3-cd)pyrene | 5.0 |
| Naphthalene | 50 |
| Phenanthrene | 4.0 |
| Pyrene | 10 |
| DFB (Surr) | 200 |
| ** This standard may be used for CCV2* | |

| PAH LEVEL 3 LINEARITY STANDARD* (5 ml of PAH level 4 linearity standard to 10 ml final volume) | |
|---|---|
| ANALYTE | ug/ml |
| Acenaphthene | 50 |
| Acenaphthylene | 25 |
| Anthracene | 1.0 |
| Benzo(a)anthracene | 2.5 |
| Benzo(b)fluoranthene | 1.0 |
| Benzo(k)fluoranthene | 1.0 |
| Benzo(g,h,i)perylene | 4.0 |
| Benzo(a)pyrene | 2.5 |
| Chrysene | 2.5 |
| Dibenz(a,h)anthracene | 10 |
| Fluoranthene | 2.5 |
| Fluorene | 5.0 |
| Indeno(1,2,3-cd)pyrene | 2.5 |
| Naphthalene | 25 |
| Phenanthrene | 2.0 |
| Pyrene | 5.0 |
| DFB (Surr) | 100 |
| ** This standard is used for CCV1* | |

| PAH LEVEL 2 LINEARITY STANDARD* (1 ml of PAH level 4 linearity standard to 10 ml final volume) | |
|---|---|
| ANALYTE | ug/ml |
| Acenaphthene | 10 |
| Acenaphthylene | 5.0 |
| Anthracene | 0.2 |
| Benzo(a)anthracene | 0.5 |
| Benzo(b)fluoranthene | 0.2 |
| Benzo(k)fluoranthene | 0.2 |
| Benzo(g,h,i)perylene | 0.8 |
| Benzo(a)pyrene | 0.5 |
| Chrysene | 0.5 |
| Dibenz(a,h)anthracene | 2.0 |
| Fluoranthene | 0.5 |
| Fluorene | 1.0 |
| Indeno(1,2,3-cd)pyrene | 0.5 |
| Naphthalene | 5.0 |
| Phenanthrene | 0.4 |
| Pyrene | 1.0 |
| DFB (Surr) | 20 |
| ** This standard may be used for CCV2* | |

| PAH LEVEL 1 LINEARITY STANDARD (1 ml of PAH level 3 linearity standard to 10 ml final volume) | |
|---|---|
| ANALYTE | ug/ml |
| Acenaphthene | 5 |
| Acenaphthylene | 2.5 |
| Anthracene | 0.1 |
| Benzo(a)anthracene | 0.25 |
| Benzo(b)fluoranthene | 0.1 |
| Benzo(k)fluoranthene | 0.1 |
| Benzo(g,h,i)perylene | 0.4 |
| Benzo(a)pyrene | 0.25 |
| Chrysene | 0.25 |
| Dibenz(a,h)anthracene | 1.0 |
| Fluoranthene | 0.25 |
| Fluorene | 0.5 |
| Indeno(1,2,3-cd)pyrene | 0.25 |
| Naphthalene | 2.5 |
| Phenanthrene | 0.2 |
| Pyrene | 0.5 |
| DFB (Surr) | 10 |

# STANDARD OPERATING PROCEDURE FOR VOLATILE ORGANICS COMPOUNDS BY EPA METHOD 624 AND SW-846 METHOD 8260B USING THE SOLATEK™ 72 AUTOSAMPLER

Originating Author: Jeff Loewe
Revision Author: Jeff Loewe

This SOP is effective upon signed approval by the following:

Unit Supervisor — Date: 11/06/01

QA/QC Director — Date: 11-6-2001

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

# 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This is a GC/MS procedure for the determination of volatile organic compounds. This procedure is applicable to the analysis of aqueous, non-aqueous liquid and solid matrix samples. The applicable compounds and their routine reporting limits are listed in section 18.0. Lower PQLs may be reported upon request from the client provided that the requested limit is above the MDL.

2.2 The equipment used in this procedure supports the application of SW-846 Method 5035. This sampling and analysis method utilizes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes.

## 3.0 SUMMARY

3.1 An aliquot of sample is withdrawn directly from the sample container (VOA vial) by the autosampler. The volatile compounds are introduced into the gas chromatograph using the purge-and-trap technique. The analytes are directly injected onto the capillary column. The column is temperature programmed to separate the analytes that are detected, using ion counts, with a mass spectrometer interfaced to a gas chromatograph. Target ions are extracted from the total ion count to identify target compounds.

3.2 Water samples are collected in glass vials with zero headspace. For low level solid samples, 5 g of sample is mixed with 10 ml of lab pure water, purged and analyzed. For medium and high level solid samples, 5 g of sample is extracted with 10 ml methanol. A portion of this extract is diluted with lab pure water, purged and analyzed.

3.3 The linear calibration range extends up to 200 ug/l.

## 4.0 DEFINITIONS

4.1 Accuracy – The degree of agreement of a measured value with the true or expected value of the quantity of concern (% recovery of a known spiked analyte).

4.2 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.3 Analyte – The specific component measured in a chemical analysis.

4.4 Analytical Batch – A group of samples which are analyzed, at the instrument level, together by a single analyst using the same method, reagents and apparatus within the same time period. Typically, these are samples in the same Run Number in the

LIMS. For purposes of quality control frequency, a batch shall not include more than 20 environmental samples.

4.5 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Field Blank – An aliquot of analyte-free water that is collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique. Also known as an equipment blank.

- Method Blank – An aliquot of analyte-free water or sand processed in an identical fashion as an environmental sample. It is a test for contamination in sample preparation and analysis. Also referred to as a Procedural Blank.

- Trip Blank – An aliquot of analyte-free water, typically supplied by the laboratory, taken to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures.

4.6 Bias – The deviation of a measured value from a known or accepted value due to matrix effects or method performance. Bias may be determined quantitatively to correct measured values. Bias may be positive or negative.

4.7 Calibration – The establishment of an analytical curve based on the absorbance, response, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type and concentration of acids, solvents, or other solutions used in the sample preparation.

4.8 Calibration Factor – A measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the volatile and semi-volatile fractions.

4.9 Continuing Calibration Verification Standard (CCV) – A standard used to verify the continued acceptability of the initial calibration curve. A continuing calibration verification must be analyzed at the beginning of each analytical sequence following an acceptable instrument tune.

4.10 Detection Limit – The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

- MDL – Method detection limit. The minimum concentration of a substance that can be measured and reported with a 99% degree of confidence. MDLs are determined by analyzing a minimum of seven consecutive standards that have been processed through all preparatory steps.

- PQL – The Practical Quantitation Limit is the lowest concentration that can reliably be achieved within specified limits of precision and accuracy during

routine laboratory operating conditions. Typically, the PQL is a value in the range of 5 - 10 times the MDL. This is the reporting limit and is also referred to as the Estimated Quantitation Limit (EQL).

4.11 Headspace – Any area in a container not completely filled by the sample, thus allowing gases to collect in that space.

4.12 Holding Time – The maximum storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

4.13 Initial Calibration Verification (ICV) – A standard used to verify the accuracy of calibration standards. Prepared from a second source than that of the calibration standards and analyzed immediately after the generation of a new calibration curve, its known value is measured against the calibration curve. This determines the integrity of working standards. Also referred to as an external verification standard or check standard.

4.14 Internal Standard (I.S.) – Applicable to ICP/MS, GC, GC/MS and LC analyses only. A compound(s), not of interest as a target compound, which is added to all samples, QC samples, and calibration standards just prior to instrument analysis. Internal standards are used as the basis for quantitation of target compounds for GC and GC/MS analysis.

4.15 Laboratory Control Sample (LCS) – An aliquot of analyte-free water or sand spiked with target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.16 Laboratory Control Sample Duplicate (LCSD) – An aliquot of analyte-free water or sand spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.17 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.18 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.19 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given

sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.20 Percent Difference (%D) – Used to compare two values, the percent difference indicates both the direction and the magnitude of the comparison. The percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

$$\%D = \frac{(X - Y)}{X} * 100$$

where: X = value 1
Y = value 2

4.21 Percent Recovery – A measure of accuracy that is calculated as the measured value relative to the true value, expressed as a percent.

$$\%R = \frac{MV}{TV} * 100$$

where: MV = measured value
TV = true value

4.22 Precision – The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the comparability of results from duplicate or replicate analyses. (%RPD between the recoveries of two known analyte spikes, and %RSD between the recoveries of three or more measurements).

4.23 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.24 Relative Percent Difference (% RPD) – Used to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

$$\%\ RPD = \frac{|X - Y|}{(X + Y) / 2} * 100$$

where: X = value 1
Y = value 2

4.25 Relative Response Factor – Applicable to GC, GC/MS, and LC analyses only. A measure of the relative response of an analyte compared to that of its internal standard. Relative response factors (RRF) are determined by analysis of

calibration standards and are used in the quantitation of target analytes in samples. RRF is calculated as follows:

$$RRF = \frac{Ax \times Cis}{Ais \times Cx}$$

where: Ax = area of the compound of interest measured
Cis = concentration of the internal standard
Ais = area of the internal standard
Cx = concentration of the analyte of interest

4.26 Relative Retention Time (RRT) – The ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_c}{RT_{is}}$$

where: $RT_c$ = Retention time for the target or surrogate in continuing calibration
$RT_{is}$ = Retention time for the internal standard in calibration standard or sample

4.27 Retention Time – The time elapsed from sample injection until the specific compound elutes or exits the chromatographic column at the detector. Each analyte has a characteristic retention time on a specific column allowing this information is used to qualitatively identify the analytes in the sample.

4.28 Sample – A portion of material supplied by the client for analysis.

4.29 Surrogate Compound – Applicable to GC, GC/MS, and LC analyses only. Compound that behaves similarly, with respect to the analytical method, as the analytes of interest but is not normally found in environmental samples. Often, surrogates are isotopic homologues of target analytes. Surrogate(s) are added to all blanks, samples and QC samples prior to preparation and analysis. Recovery of surrogates is used to assess method performance.

4.30 Tune – Analysis of a compound to verify the operating conditions of the instrument by comparing the ion abundance of various masses.

## 5.0 INTERFERENCES

5.1 Chemical interference is minimized through the use of ion counts and internal standards.

5.2 Occasionally, samples may foam while being purged. This problem can be corrected through dilution or the use of a silicon anti-foaming agent.

5.3 Contamination may occur from impurities in the purge gas, carryover from high-level samples or environmental contamination resulting from the introduction of target analytes into the lab. Method blanks are used to verify that the system is

clean and suitable for analysis. Analyzing additional blanks and/or baking out the column between analyses can eliminate carryover contamination.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 Various gas-tight syringes including 10, 25 and 50 ul

7.3 5 ml gas-tight syringe with Luer-lock tip

7.4 2 ml autosampler vials with crimp tops or screw caps

7.5 1 ml miniinert vessels with caps

7.6 40 ml glass VOA vials with Teflon lined screw cap septa

7.7 HP 5890 Gas Chromatograph system including a Tekmar SOLAtek 72 autosampler, Tekmar LSC3000 purge and trap unit, and a HP 5970 Mass Selective Detector (or equivalent system)

7.8 Chromatography column: DB-624, J&W Scientific (catalog #123-1334) or equivalent. Length = 30 m, ID = 0.32 mm, film thickness = 1.8 um.

7.9 Vocarb 3000 Trap: Supelco Purge Trap K (catalog #24920-U) or equivalent

7.10 Computer with MS Chemstation software, monitor, and printer

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

8.2.1 Analyte-free water: Prepare by continuously bubbling nitrogen gas through tap water.

8.2.2 Methanol, purge and trap grade

8.2.3 Sand: Prepare by baking in an oven at >70°C for a minimum of 3 hours.

8.3 Standards

8.3.1 Stock Internal Standard (I.S.), 2000 ug/ml each in methanol.

| VENDOR | CAT # | COMPOUNDS |
|---|---|---|
| Accustandard | M-8260A/B-IS-10X | Chlorobenzene-d5; 1,4-Dichlorobenzene-d5; Fluorobenzene |

8.3.2 Stock Surrogate (SURR) Standard, 2000 ug/ml each in methanol.

| VENDOR | CAT # | COMPOUNDS |
|---|---|---|
| Accustandard | M-8260A/B-SS-10X | 4-Bromofluorobenzene; Dibromofluoromethane; 1,2-Dichloroethane-d5; Toluene-d8 |

8.3.3 Working I.S./SURR Solution, 50 ug/ml each: In a 10 ml volumetric flask, dilute 250 ul of Stock I.S. and Stock SURR standards to the mark with methanol. Store the solution in the freezer. Prepare new solution at a minimum of every 6 months. The addition of 5 ul of this solution to 5 ml of sample yields a concentration of 50 ug/l.

8.3.4 Stock Calibration Standards, 2000 ug/ml each (unless otherwise noted) in methanol.

| GROUP | VENDOR | CAT # | COMPOUNDS |
|---|---|---|---|
| Non-gases | Accustandard | M-502A-R2-10X | See Table in Section 18.0 |
| Method 8260 Additions | Accustandard | M-8260-ADD-10X | Acetone; 2-Butanone; Carbon disulfide; 2-Chloroethylvinyl ether; 2-Hexanone; Iodomethane; 4-Methyl-2-pentanone; Vinyl acetate |
| Gases | Accustandard | M-502B-10X | Bromomethane; Chloroethane; Chloromethane; Dichlorodifluoromethane; Trichlorofluoromethane; Vinyl chloride |
| | Accustandard | M-603-10X | Acrolein; Acrylonitrile @ 10,000 ug/ml each |
| | Accustandard | APP-9-005-10X | Acetonitrile @ 10,000 ug/ml |
| | Accustandard | S-078-10X | Methyl-t-butyl ether |

8.3.5 Intermediate Calibration Standards: In separate 1 ml volumetric flasks, prepare the following dilutions of the stock calibration standards with methanol. Store the standards in a freezer. Prepare new solutions at a minimum of every 6 months with the exception of the Gases, which must be prepared on a weekly basis.

| Group | Vol. Stock Cal. Std., ul | Final Conc., ug/ml |
|---|---|---|
| Non-gases and Method 8260 Additions | 25 each | 50 |
| Gases | 25 | 50 |
| Acrolein; Acrylonitrile | 50 | 500 |
| Acetonitrile | 50 | 500 |
| Methyl-t-butyl ether | 25 | 50 |

8.3.6 Working Calibration Standards: Water Linearity - In separate 50 ml volumetric flasks, prepare the following dilutions with lab pure water. Transfer the prepared standards to 40ml VOA vials leaving zero headspace.

| Linearity Standard # | Vol. Inter. Cal. Std, ul | Final Conc. ug/l |
|---|---|---|
| 1 | 5 | 5 |
| 2 | 10 | 10 |
| 3 | 20 | 20 |
| 4 | 50 | 50 |
| 5 | 100 | 100 |
| 6 | 200 | 200 |

Soil Linearity -- Using a 5.0 ml syringe, individually prepare the following dilutions with lab pure water. Transfer the prepared standards to 40ml VOA vials for analysis.

| Linearity Standard # | Vol. Inter. Cal. Std, ul | Final Conc. Ug/l |
|---|---|---|
| 1 | 0.5 | 5 |
| 2 | 1 | 10 |
| 3 | 2 | 20 |
| 4 | 5 | 50 |
| 5 | 10 | 100 |
| 6 | 20 | 200 |

8.3.7 Working Calibration Verification (CCV) Standard, 50 ug/l each: Use the Linearity Standard #4.

8.3.8 Stock Verification/Spike Standards, 2000 ug/ml each (unless otherwise noted) in methanol.

| GROUP | VENDOR | CAT # | COMPOUNDS |
|---|---|---|---|
| Non-gases | Supelco | 502111 | See Table in Section 18.0 |
| Gases | Supelco | 48799-U | Bromomethane; Chloroethane; Chloromethane; Dichlorodifluoromethane; Trichlorofluoromethane; Vinyl chloride |
| 8240B Cal Mix 2 | Supelco | 47364 | Acetone, Acetonitrile, Acrylonitrile, 2-Butanone, 2-Hexanone, 4-methyl-2-pentanone |
| MTBE | Restek | 30402 | Methyl-t-butyl ether |
| Carbon Disulfide | Restek | 30258 | Carbon Disulfide |
| 2-CEVE | Restek | 30265 | 2-Chloroethylvinyl ether |
| Vinyl Acetate | Restek | 30216 | Vinyl Acetate |

8.3.9 Intermediate Verification/Spike Standards: In separate volumetric flasks, prepare the following dilutions of the stock calibration verification standards with methanol. Store the standards in a freezer. Prepare new solutions at a minimum of every 6 months with the exception of the Gases, which must be prepared on a weekly basis.

| Group | Vol. Stock Verif. Std., ul | Final Vol., ml | Final Conc., ug/ml |
|---|---|---|---|
| Gases | 25 | 1 | 50 |
| Non-gases | 25 | 1 | 50 |
| Method 8240B Cal Mix 2 | 25 | 1 | 50 |
| MTBE, Carbon Disulfide, 2-CEVE, and Vinyl Acetate | 25 each | 1 | 50 |

8.3.10 Initial Calibration Verification (ICV) Standard, 50 ug/l: For water calibrations, in a 50 ml volumetric flask, dilute 50 ul of the intermediate verification/spike standards to the mark with lab pure water. For solid calibrations, in a 5.0 ml syringe, dilute 5.0 ul of the intermediate verification/spike standards to volume with lab pure water. Transfer this prepared standard to a 40ml VOA vial for analysis.

8.3.11 LCS: For water calibrations, in a 50 ml volumetric flask, add 20 ul of the intermediate verification/spike standards to 50 ml of lab pure water. This prepares a LCS on 20 ug/l each. For a solid LCS, add 5.0 ul of the intermediate verification/spike standards to 5 ml of lab pure water. Transfer this standard to a 40ml VOA vial containing 5g of sand. This prepares a LCS of 50 ug/l each.

8.3.12 MS/MSD, 50 ug/l each for Method 8260 and 20 ug/l each for Method 624: In a 25 ml volumetric flask, measure 25 ml of sample and add 25 ul of the intermediate verification/spike standards to prepare a MS of 50 ug/l. Use 10 ul of the intermediate verification/spike standards to prepare a MS of 20 ug/l. For solid samples, spike 5g of sample with 5.0 ul the standards for Method 8260. This prepares a spike of 50 ug/kg each.

8.3.13 Method Blank (MB): Analyte-free water is used for the MB associated with the analysis of water and methanol extracted samples. 5g of sand is used for the MB associated with the analysis of solid samples.

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Water samples should be collected in a 40 ml glass vial with a Teflon lined lid (VOA vial) leaving zero headspace. Preservation consists of HCl to pH < 2 and storage in the range of 0.1-6°C. Samples are stored in the dedicated VOA cooler and analysis must be performed within the maximum allowable hold time of 14 days from collection for acid preserved water samples. The analyst must measure the pH after sample analysis has been performed. The result of these checks is noted on the injection log. Any sample not meeting the requirement of pH < 2 must be qualified in a Case Narrative to the client.

9.3 Solid samples should be collected according to the specifications described below. The collection steps include the preservation technique. All sample containers received for analysis according to Method 5035 for strict adherence to the method must be chemically preserved with sodium bisulfate ($NaHSO_4$) or methanol and retained at 4°C until analysis. The maximum allowable hold time for chemically preserved samples is 14 days from collection. Samples received for programs that allow freezing as an alternative to the chemical preservation must be frozen at –12 ± 2°C within 24 hours of receipt at the lab to inhibit biodegradation. This thermal preservation technique provides for a maximum hold time of 7 days from collection. Without the thermal preservation (i.e. freezing), samples must be analyzed within 48 hours of collection. Samples stored at 4°C are retained in the dedicated VOA cooler and samples stored at –12°C are retained in the organics freezer.

9.3.1 Method 5035

9.3.1.1 Low level samples. At the time of sample collection, 5g of sample are placed into a pre-weighed VOA vial. The vials are sealed, chilled to 4°C, and shipped to the laboratory for receipt within 48 hours of collection. Upon receipt, the vials are re-weighed to obtain the weight of sample, which is recorded in a logbook.

9.3.1.2 Medium/High level samples. At the time of sample collection, 5g of sample are placed into a pre-weighed VOA vial containing 10 ml of purge-and-trap grade methanol. The vials are sealed, chilled to 4°C, and shipped to the laboratory. Upon receipt, the vials are re-weighed to obtain the weight of sample, which is recorded in a logbook.

9.3.1.3 Field-unpreserved samples. The sample is collected in an air-tight storage container. These devices collect the sample in a storage chamber that may be sealed leaving zero headspace. Acceptable examples include Encore samplers or other coring devices. The samples are then chilled to 4°C and shipped to the laboratory for receipt within 48 hours of collection.

9.3.2 Method 5030

9.3.2.1 Field-unpreserved samples. The sample is collected in container with minimal headspace and unopened until analysis. Acceptable examples include glass jars with Teflon lined lids. The samples are then chilled to 4°C and shipped to the laboratory for receipt within 48 hours of collection.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Analyze four separate standards prepared in the range of 8-10 times the method detection limit listed in section 18.0. These standards must be from a source different from that used for calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.2 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Analyze a minimum of seven (maximum of ten) standards prepared in the range of 2-5 times the method detection limit listed in section 18.0 or an estimated detection limit. These standards must be taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.3 *Internal Standards* (I.S.) must be added to all standards, QC samples and environmental samples. Acceptance criteria for CCV are RT + 30 seconds from that in the midpoint level standard of the most recent initial calibration and area counts within the range of -50 to +100% of those in the same linearity standard. These criteria are evaluated by the data system and printed on the Continuing Calibration Report. Failures are automatically flagged on this report. If acceptance criteria are not met for the CCV, perform instrument maintenance or perform a new initial calibration. As there are no absolute requirements for I.S. recovery in samples, the acceptance criteria for the CCV should be used as guidance in evaluating the response for samples. Typically, if the I.S. exhibits poor response the surrogates will not meet their QC criteria, which will result in sample reanalysis. Therefore, if the sample internal standard response does not meet the CCV acceptance criteria evaluate the surrogate recoveries. If the surrogate recoveries are acceptable and the internal standard response for the sample is in the range of –75 to +100% the sample results should be reported. If the surrogate recoveries are acceptable and the internal standard response for the sample is below 25% the analyst should consult with the Unit Supervisor and use discretion on whether to report or reanalyze the sample. This discretion is dependent on the experience of the analyst and their Supervisor and factors such as a consistent response for the I.S. should be considered. If the surrogate recoveries are not acceptable and the internal standard response in the sample does not meet the CCV acceptance criteria, the sample should be reanalyzed regardless of the hold time. If reanalysis yields poor internal standard response, or if reanalysis is performed beyond the hold time, both sets of data should be reported to the client and a Case Narrative written.

10.4 *Surrogate* (SURR) compounds must be added to all quality control samples, blanks, and samples. Acceptance criteria are the statistically generated matrix-specific recovery limits listed in section 18.0. These criteria are evaluated by the LIMS and printed on the LIMS QC Report and analytical report submitted to the client. Failures are automatically flagged with a "S" on these reports. If the acceptance criteria are not met for a sample, reanalyze. If reanalysis does not

yield acceptable recovery, both sample sets must be supplied to the client. If reanalysis is performed beyond the maximum allowable hold time, both sample sets must be supplied to the client and the appropriate result flagged with a "H" qualifier as defined in the LIMS. If insufficient sample is available for reanalysis the original result should be reported and qualified in the Case Narrative to state this as the reason for no reanalysis being performed. If the acceptance criteria are not met for a method blank but are met for the QC samples and environmental samples, report the sample results qualified for the MB failure in the Case Narrative.

10.5 An *Instrument Tune* must be performed at the beginning of every 12 hour analytical sequence. This is accomplished by injecting 1 ul of the working I.S./SURR solution into the injection port of the GC while pressing the start button to initiate the run using the BFB method programmed into the software. Acceptance criteria are listed in section 18.0. If the acceptance criteria are not met, repeat. Sample analysis cannot be performed without first meeting the tune criteria.

10.6 An *Initial Calibration Verification (ICV)* must be analyzed immediately after the initial linearity (ICAL). This is the analysis of a second source standard. Acceptance criteria are the statistically generated recovery limits listed in section 18.0. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, analysis must be stopped, the problem corrected, and the instrument recalibrated.

10.7 A *Continuing Calibration Verification (CCV)* standard is a calibration source standard and must be analyzed at the beginning of each analytical sequence following an acceptable instrument tune. The 12-hour analytical sequence begins with the injection of BFB, continues through the analysis of samples and QC samples. Acceptance criteria for Method 8260 are RF for SPCCs (see section 11) and RF for CCCs $\leq$ 20% difference from the initial calibration. If acceptable criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, recalibrate. Samples with a non-detect concentration may be reported if the CCV fails to meet the acceptance criteria with a positive bias.

10.8 A *Method Blank* (MB) must be analyzed with each batch of up to 20 samples per day (at a minimum of 1 per day) per matrix and with each new set of reagents. Analyte-free water or sand is used, as appropriate. Acceptance criteria are no detects above the PQL. If the MB concentration is greater than the PQL, the MB is acceptable if the blank concentration is less than 1/10 of the sample concentration or if there are no detects in the sample. If the acceptance criteria are not met, reanalyze. If this reanalysis does not meet the acceptance criteria, those samples that are effected by the failed MB must be reanalyzed. Samples that are valid with the failed MB may be reported with a "B" qualifier as defined in the LIMS. When a MB is contaminated, corrective action steps must be taken to identify and eliminate the cause of the contamination.

10.9 A *Laboratory Control Sample (LCS)* must be analyzed at the beginning of each batch of a maximum 20 environmental samples per matrix immediately following an acceptable method blank and prior to the analysis of any environmental

samples. This is the analysis of a second source standard spiked into analyte-free water or sand, as appropriate, and serves as the daily calibration check as required in Method 624. Acceptance criteria are the statistically generated recovery limits listed in section 18.0 for Method 8260B or the recovery limits in Table 5 of Method 624, as appropriate. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, analysis must be stopped, the problem corrected, and the instrument recalibrated. If samples are analyzed in a sequence with a failed LCS, the samples must be reanalyzed. If insufficient sample is available for reanalysis the original result should be reported and qualified in the Case Narrative. If the hold time has expired and reanalysis performed, both sets of data should be reported and the appropriate result flagged with a "H" qualifier as defined in the LIMS. Samples with a non-detect concentration may be reported if the LCS fails to meet the acceptance criteria with a positive bias. A full-list spike is added for the MS/MSD, although acceptance is based upon only those compounds in Table 5 of Method 624 (see tables in section 18.0). The recoveries of the other compounds are evaluated as informational only or as required by the request of the client.

10.10 A *Matrix Spike and Matrix Spike Duplicate* (MS/MSD) must be analyzed with each batch of up to 20 samples per day (at a minimum of 1 per day). When insufficient sample is available for a MSD, a MS may be performed on two different samples or a duplicate laboratory control sample (LCSD) should be analyzed. Accuracy criteria are the statistically generated recovery limits listed in section 18.0 for Method 8260B or the recovery limits in Table 5 of Method 624, as appropriate. The MS/MSD remains acceptable if the failed recovery is positive bias (high) and there are no detects in the sample. If the acceptance criteria are not met for the MS, evaluate the MSD for accuracy. If the acceptance criteria are met in the MSD, continue. If the accuracy criteria are not met in the MS or MSD, and the LCS is in control, assume matrix interference and report the results with a "S" qualifier as defined in the LIMS. Precision criteria are the statistically generated limits in the LIMS. If the precision criteria are not met, report the results with a "R" qualifier as defined in the LIMS. A full-list spike is added for the MS/MSD, although acceptance is based upon only those compounds in Table 5 of Method 624 (see tables in section 18.0). The recoveries of the other compounds are evaluated as informational only or as required by the request of the client.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Perform the required preventative maintenance as necessary. Daily, the carrier gas must be checked. A new tank is used when the pressure drops below 500 psi. A new Initial Calibration (linearity) is required as indicated by the quality control elements.

11.2 Instrument conditions are as follows:

Concentrator

| | |
|---|---|
| Standby: | 35°C |
| Purge: | 11 minutes |
| Dry Purge: | 6 minutes |
| Desorb preheat: | 245°C |
| Desorb: | 2 minutes at 250°C |
| Bake: | 10 minutes at 260°C |

Gas Chromatograph

| | |
|---|---|
| Inlet B: | 200°C |
| Detector B: | 250°C |

BFBTune.m

| | |
|---|---|
| Initial temperature: | 35°C |
| Initial time: | 2.0 minutes |
| Rate: | 20°C / minute |
| Final temperature: | 195°C |
| Final time: | 0.0 minutes |
| Run time: | 10.0 minutes |

NEWVOA.m

| | |
|---|---|
| Initial temperature: | 35°C |
| Initial time: | 2.0 minutes |
| Rate A: | 8°C / minute |
| Final temperature: | 130°C |
| Final time: | 0.0 minutes |
| Rate B: | 15°C / minute |
| Final temperature: | 180°C |
| Final time: | 0.0 minutes |
| Rate C: | 25°C / minute |
| Final temperature: | 230°C |
| Final time: | 4.0 minutes |
| Run time: | 23.2 minutes |

11.3 Purging and transfer to the LSC3000 is initiated through the SOLAtek autosampler. The GC/MS is enabled by using the Method – Run pulldown menus on the instrument top window on the computer. The internal standards and surrogates are automatically added to each standard/sample.

11.4 Separate 6-point calibrations (linearity) are performed for waters and solids. The solids linearity uses a heated purge. The concentration of the low standard must be set near or below the routine PQL for each compound.

11.5 Set up analysis sequence in Sample Table Log in *Sequence* in main menu.

11.6 Enter the sequence as it is to be run, including the applicable analysis method. An ICV standard must be analyzed following the generation of a new linearity and the acceptance criteria met before continuing with sample analysis. If the acceptance

criteria are met, rename the method "WTRmmdd" or "SOILmmdd", as appropriate, where mmdd designates the month and date of the new linearity.

11.7 Following an acceptable ICV, a BFB tune, CCV, method blank and LCS must be run. Samples may be analyzed if these controls are acceptable.

11.8 After each sample has run, the report is retrieved and quantitated against the current initial linearity. The chromatogram is examined to determine that all compounds present have been detected.

11.9 For standards, the software will calculate RF values and % difference values for all analytes. The average RF must be < 30% for the CCCs and <15% for all target compounds as well as the SPCCs must meet the criteria below. Where these criteria are met the calibration is considered linear and the average RF is used for concentration calculations.

SPCC Criteria
≥ 0.300 for Chlorobenzene and 1,1,2,2-Tetrachloroethane
≥0.100 for Chloromethane, and 1,1-Dichloroethane, and Bromoform

CCC Compounds
1,1-Dichloroethene, 1,2-Dichloropropane, Chloroform, Ethylbenzene, Toluene and Vinyl chloride

11.9.1 If the average RF > 15 for any target compound, averaging may be used to identify a linear curve. This technique assesses the average %RSD of all compounds in the calibration curve. If the RSD of all (target and non-target) compounds is ≤ 15%, the calibration can be considered acceptable and the average RF used. When used, the fact of its use and average RSD must be reported to the data user.

11.10 If the linearity requirements are not met, take appropriate corrective actions and recalibrate. Analysis of environmental samples cannot proceed without the generation of an acceptable linearity.

## 12.0 PROCEDURE

12.1 SAMPLE PREPARATION

12.1.1 Water Samples

12.1.1.1 Load the VOA vials onto the autosampler. The purge and trap unit removes a 5 ml aliquot of sample and adds 5ul of the internal standard/surrogate solution for analysis. Dilutions up to 1:10 can be programmed and prepared by the autosampler. Higher dilutions, if necessary, must be prepared in volumetric flasks and transferred to VOA vials for analysis.

12.1.2 Solid Samples – Method 5035

12.1.2.1 For **low-level samples collected directly into VOA vials** - Load the VOA vials onto the autosampler. The vials contain a stir bar and 5g of sample and the $NaHSO_4$ preservative (if used). The purge and trap unit adds 5ul of the internal standard/surrogate solution and 10 ml lab pure water for analysis.

12.1.2.2 For **low-level samples collected with coring devices** – On the day of analysis, allow the unopened storage device to reach ambient temperature, extrude the sample into a tared VOA vial containing a stir bar and record the sample weight. Record the weight in the logbook and place the vial on the autosampler. The purge and trap unit adds 5ul of the internal standard/surrogate solution and 10 ml lab pure water for analysis.

12.1.2.3 For **medium/high level samples collected with methanol preservation** – Transfer 1 ml of the extract to a 50 ml volumetric flask partially filled with lab pure water. (If the sample is expected to be very high in concentration use a lesser sample volume than 1 ml as the maximum sample : water ratio is 100 ul sample per 5.0 ml water.) Dilute the sample to the mark with lab pure water, transfer the solution to a VOA vial and load the vial onto the autosampler. Analyze the sample as a water sample (i.e. non-heated purge). The purge and trap unit adds 5ul of the internal standard/surrogate solution for analysis.

12.1.3 Solid Samples – Method 5030

12.1.3.1 For **low-level samples** - Transfer 5.0g of sample into a tared VOA vial containing a stir bar and record the sample weight. Load the VOA vial onto the autosampler. The purge and trap unit adds 5ul of the internal standard/surrogate solution and 10 ml lab pure water for analysis.

12.1.3.2 For **medium/high level samples** – Transfer 5.0g of sample into a tared VOA vial containing a stir bar and record the sample weight. Add 10.0 ml of methanol into the vial, cap and shake for 2 minutes. Transfer approximately 2 ml of the extract into a GC autosampler vial. This extract must be stored in the dark, at 4°C, until analyzed. To analyze, transfer a measured aliquot (maximum of 1 ml) of the extract to a 50 ml volumetric flask and dilute to the mark with analyte-free water. (This corresponds to the method required maximum of 100 ul sample per 5 ml final volume.) Transfer the diluted extract into a VOA vial leaving zero headspace. Place the vial on the autosampler and analyze as a water sample (i.e. non-heated purge).

12.2 ANALYSIS

12.2.1 Once it has been determined that the tune and calibration verifications meet their respective criteria, the analysis of samples can begin. A typical sequence follows the order: Tune, CCV, MB, LCS, environmental samples and QC samples.

12.2.2 If the concentration of any target compound in a sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and analyzed.

12.2.2.1 For water samples, dilutions should be prepared in volumetric flasks. Intermediate dilutions may be necessary for extremely large dilutions.

12.2.2.2 For solid samples, as little as 0.5g of sample may be weighed and analyzed. If a greater dilution is required, the medium level, methanol extract, method must be used.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 After review, enter final results into the LIMS system. Results flagged by the LIMS with an "E" qualifier are above the linear range of the instrument. There is less certainty in these data and, if sufficient sample and holding time are available, should be reanalyzed at an appropriate dilution. Details on the procedure for entering analytical data are in the Data Entry SOP. The peak integrations must be performed according to the Manual Integration of Chromatographic Peaks SOP.

13.2 The data system calculates the sample concentration as follows:

$$\text{Conc.} = \frac{(A_x)(I_s)(DF)}{(A_{IS})(RF)(V_o)}$$

Where: $A_x$ = Area of characteristic ion for compound being measured
$I_s$ = Amount of internal standard injected (ng)
DF = dilution factor
$A_{IS}$ = Area of characteristic ion for the internal standard
RF = Initial average response factor for compound being measured
$V_o$ = Volume of water purged (ml) or mass of soil purged (g)
NOTE: $(A_x)(I_s) / (A_{IS})(RF)$ is calculated by the computer.

13.3 The LIMS calculates the dry-weight concentration for solid samples as follows:

$$\text{Conc. Dry} = \frac{(\text{wet weight conc.})}{(100 - \%\ \text{Moisture})}$$

13.4 The data system software evaluates the retention time and comparison to the characteristic ions to identify any compounds present. The characteristic ions of the reference spectrum are the three ions of greatest intensity (or any ions having a relative intensity greater than 30% if less than three ions are present). The following criteria are used for qualitative identification.

13.4.1 The characteristic ions of a compound must have a relative retention time of ± 0.06 units of the standard (RT ± 30 sec for Method 624).

13.4.2 The relative intensities of the characteristic ions are within 20% of those ions in the reference spectrum (30% for Method 8260).

13.4.3 Structural isomers having a resolution of < 25% are considered isomeric pairs.

## 14.0 METHOD PERFORMANCE

14.1 The latest Method Detection Limit Study and Initial Demonstration of Capability Study data are listed in section 18.0.

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Refer to the SIMALABS International Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, extract or standard.

## 17.0 REFERENCES

17.1 USEPA Method 624

17.2 SW-846 Method 8260B

17.3 SW-836 Method 5030

17.4 SW-846 Method 5035

17.5 SIMALABS International Quality Assurance Plan, current revision

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

| Calibration Standard Compounds | |
|---|---|
| M-502A-R2-10X<br><br>Non-gases | Benzene; Bromobenzene; cis-1,3-Dichloropropene; Bromodichloromethane; Bromoform; n-Butylbenzene; sec-Butylbenzene; tert-Butylbenzene; Carbon tetrachloride; Chlorobenzene; Chloroform; 2-Chlorotoluene; 4-Chlorotoluene; Dibromochloromethane; 1,2-Dibromoethane; Dibromomethane; 1,2-Dibromo-3-chloropropane; 1,2-Dichlorobenzene; 1,3-Dichlorobenzene; 1,4-Dichlorobenzene; 1,1-Dichloroethane; 1,2-Dichloroethane; 1,1-Dichloroethene; cis-1,2-Dichloroethene; ; trans-1,2-Dichloroethene; 1,2-Dichloropropane; 1,3-Dichloropropane; 2,2-Dichloropropane; 1,1-Dichloropropene; trans-1,3-Dichloropropene; Ethylbenzene; Hexachlorobutadiene; Isopropylbenzene; p-Isopropyltoluene; Methylene chloride; Naphthalene; n-Propylbenzene; Styrene; 1,1,1,2-Tetrachloroethane; 1,1,2,2-Tetrachloroethane; Tetrachloroethene; Toluene; 1,1,1-Trichloroethane; 1,1,2-Trichloroethane; Trichloroethene; 1,2,3-Trichlorobenzene; 1,2,4-Trichlorobenzene; 1,2,3-Trichloropropane; 1,2,4-Trimethylbenzene; 1,3,5-Trimethylbenzene; o-Xylene; m-Xylene; p-Xylene |

| ICV/LCS/MS/MSD (2nd source verification) Standard Compounds | |
|---|---|
| Supelco 502111<br><br>Non-gases | Benzene; Bromobenzene; cis-1,3-Dichloropropene; Bromodichloromethane; Bromoform; n-Butylbenzene; sec-Butylbenzene; tert-Butylbenzene; Carbon tetrachloride; Chlorobenzene; Chloroform; 2-Chlorotoluene; 4-Chlorotoluene; Dibromochloromethane; 1,2-Dibromoethane; Dibromomethane; 1,2-Dibromo-3-chloropropane; 1,2-Dichlorobenzene; 1,3-Dichlorobenzene; 1,4-Dichlorobenzene; 1,1-Dichloroethane; 1,2-Dichloroethane; 1,1-Dichloroethene; cis-1,2-Dichloroethene; ; trans-1,2-Dichloroethene; 1,2-Dichloropropane; 1,3-Dichloropropane; 2,2-Dichloropropane; 1,1-Dichloropropene; trans-1,3-Dichloropropene; Ethylbenzene; Isopropylbenzene; p-Isopropyltoluene; Methylene chloride; Naphthalene; n-Propylbenzene; Styrene; 1,1,1,2-Tetrachloroethane; 1,1,2,2-Tetrachloroethane; Tetrachloroethene; Toluene; 1,1,1-Trichloroethane; 1,1,2-Trichloroethane; Trichloroethene; 1,2,3-Trichlorobenzene; 1,2,4-Trichlorobenzene; 1,2,3-Trichloropropane; 1,2,4-Trimethylbenzene; 1,3,5-Trimethylbenzene; o-Xylene; m-Xylene; p-Xylene |

| Method 624 / 8260B Tune Criteria | |
|---|---|
| Mass | Ion Abundance Criteria |
| | |
| 50 | 15 – 40% of mass 95 |
| | |
| 75 | 30 – 60 % of mass 95 |
| | |
| 95 | Base peak, 100% relative abundance |
| 96 | 5 – 9 % of mass 95 |
| | |
| 173 | < 2 % of mass 174 |
| 174 | > 50 % of mass 95 |
| 175 | 5 – 9 % of mass 174 |
| 176 | > 95 but < 101 % of mass 174 |
| 177 | 5 – 9 % of mass 176 |

| MDL Study Results and PQLs | | |
|---|---|---|
| Analyte | MDL, ug/l or ug/kg | PQL, ug/l or ug/kg |
| Acetone | 3.89 | 100 |
| Acrylonitrile | 2.95 | 100 |
| Benzene | 0.87 | 5 |
| Bromodichloromethane | 1.18 | 5 |
| Bromoform | 0.67 | 5 |
| Bromomethane | 6.17 | 10 |
| 2-Butanone | 9.24 | 10 |
| Carbon Disulfide | 1.05 | 5 |
| Carbon tetrachloride | 1.07 | 5 |
| Chlorobenzene | 0.72 | 5 |
| Chloroethane | 7.66 | 10 |
| Chloroform | 0.92 | 5 |
| Chloromethane | 2.96 | 10 |
| Dibromochloromethane | 0.71 | 5 |
| 1,1-Dichloroethane | 2.28 | 5 |
| 1,2-Dichloroethane | 0.84 | 5 |
| 1,1-Dichloroethene | 1.59 | 5 |
| cis-1,2-Dichloroethene | 1.27 | 5 |
| trans-1,2-Dichloroethene | 2.34 | 5 |
| 1,2-Dichloropropane | 1.44 | 5 |
| cis-1,3-Dichloropropene | 1.17 | 5 |
| trans-1,3-Dichloropropene | 0.76 | 5 |
| Ethylbenzene | 0.64 | 5 |
| 2-Hexanone | 6.48 | 10 |
| 4-Methyl-2-Pentanone | 1.44 | 5 |
| Methyl-t-Butyl Ether | 1.24 | 5 |
| Methylene chloride | 1.85 | 5 |
| Styrene | 1.03 | 5 |
| 1,1,1,2-Tetrachloroethane | 0.62 | 5 |
| 1,1,2,2-Tetrachloroethane | 1.10 | 5 |
| Tetrachloroethene | 1.16 | 5 |
| Toluene | 1.68 | 5 |
| 1,1,1-Trichloroethane | 1.09 | 5 |
| 1,1,2-Trichloroethane | 1.41 | 5 |
| Trichloroethene | 0.87 | 5 |
| Trichlorofluoromethane | 1.82 | 10 |
| Vinyl chloride | 1.66 | 10 |
| m,p-Xylene | 2.26 | 10 |
| o-Xylene | 0.93 | 5 |
| 1,1-Dichloropropene | 1.17 | 5 |
| 1,2,4-Trichlorobenzene | 1.20 | 5 |
| 1,2,4-Trimethylbenzene | 1.02 | 5 |
| 1,2-Dibromoethane | 1.24 | 5 |
| 1,2-Dichlorobenzene | 0.65 | 5 |
| 1,3,5-Trimethylbenzene | 1.05 | 5 |
| 1,3-Dichlorobenzene | 0.86 | 5 |
| 1,3-Dichloropropane | 1.24 | 5 |
| 1,4-Dichlorobenzene | 0.78 | 5 |
| 2-Chloroethyl vinyl ether | 1.20 | 10 |
| 2-Chlorotoluene | 1.64 | 5 |
| 4-Chlorotoluene | 1.03 | 5 |
| Acetonitrile | 9.79 | 100 |
| Bromobenzene | 0.91 | 5 |
| Dibromomethane | 0.76 | 5 |
| Dichlorodifluoromethane | 15.82 | 10 |
| Isopropylbenzene | 1.40 | 5 |
| n-Butylbenzene | 1.28 | 5 |
| n-Propylbenzene | 1.23 | 5 |
| p-Isopropyltoluene | 0.95 | 5 |
| sec-Butylbenzene | 0.81 | 5 |
| tert-Butylbenzene | 0.93 | 5 |

## Surrogate Recovery Limits - Water

| | 5ml Purge | | 25ml Purge | |
|---|---|---|---|---|
| Analyte | Low | High | Low | High |
| 1,2-Dichloroethane-d4 | 77.5 | 120 | 80.9 | 118 |
| 4-Bromofluorobenzene | 82.6 | 112 | 85.3 | 109 |
| Dibromofluoromethane | 81.3 | 117 | 91.6 | 109 |
| Toluene-d8 | 86.9 | 113 | 87.3 | 111 |

## Surrogate Recovery Limits - Solid

| | 5030 | | 5035 | |
|---|---|---|---|---|
| Analyte | Low | High | Low | High |
| 1,2-Dichloroethane-d4 | 67.9 | 142 | 87.5 | 136 |
| 4-Bromofluorobenzene | 58.6 | 119 | 60.6 | 120 |
| Dibromofluoromethane | 70.8 | 132 | 85.8 | 123 |
| Toluene-d8 | 72.7 | 141 | 75.4 | 142 |

## Method 624/8260 ICV Recovery Limits

| Analyte | Low | High |
|---|---|---|
| Chloromethane | 5.7 | 164 |
| Vinyl chloride | 37.2 | 152 |
| Bromomethane | 0 | 287 |
| Chloroethane | 0 | 347 |
| Trichlorofluoromethane | 61.4 | 154 |
| 1,1-Dichloroethene | 56.6 | 124 |
| Methylene chloride | 50.7 | 136 |
| trans-1,2-Dichloroethene | 59.9 | 133 |
| 1,1-Dichloroethane | 64.3 | 124 |
| Chloroform | 75.8 | 116 |
| 1,2-Dichloroethane | 73.3 | 126 |
| 1,1,1-Trichloroethane | 72.1 | 129 |
| Benzene | 74.1 | 119 |
| Carbon tetrachloride | 56.6 | 142 |
| 1,2-Dichloropropane | 75.4 | 121 |
| Trichloroethene | 70.7 | 127 |
| Bromodichloromethane | 71.9 | 122 |
| 2-Chloroethyl vinyl ether | 10 | 197 |
| 1,1,2-Trichloroethane | 75.6 | 123 |
| Dibromochloromethane | 68.1 | 130 |
| Bromoform | 54.9 | 138 |
| cis-1,3-Dichloropropene | 74.2 | 128 |
| trans-1,3-Dichloropropene | 75.8 | 127 |
| Toluene | 79.6 | 122 |
| Tetrachloroethene | 71.7 | 139 |
| Chlorobenzene | 81.6 | 122 |
| Ethylbenzene | 71.2 | 140 |
| 1,1,2,2-Tetrachloroethane | 54.8 | 144 |
| 1,3-Dichlorobenzene | 73.8 | 113 |
| 1,4-Dichlorobenzene | 78.9 | 119 |
| 1,2-Dichlorobenzene | 79.6 | 121 |

## Method 624 LCS, MS, MSD Recovery Limits

| Analyte | Low | High | RPD |
|---|---|---|---|
| Chloromethane | 0 | 273 | 90.9 |
| Vinyl chloride | 0 | 251 | 31.2 |
| Bromomethane | 0 | 242 | 52.8 |
| Chloroethane | 14 | 230 | 29.3 |
| Trichlorofluoromethane | 17 | 181 | 81.6 |
| 1,1-Dichloroethene | 0 | 234 | 31 |
| Methylene chloride | 0 | 221 | 28.7 |
| trans-1,2-Dichloroethene | 54 | 156 | 66.1 |
| 1,1-Dichloroethane | 59 | 155 | 14.2 |
| Chloroform | 51 | 138 | 25 |
| 1,2-Dichloroethane | 49 | 155 | 23.3 |
| 1,1,1-Trichloroethane | 52 | 162 | 29 |
| Benzene | 37 | 151 | 23.7 |
| Carbon tetrachloride | 70 | 140 | 31.1 |
| 1,2-Dichloropropane | 0 | 210 | 26 |
| Trichloroethene | 71 | 157 | 26.9 |
| Bromodichloromethane | 35 | 155 | 28.4 |
| 2-Chloroethyl vinyl ether | 0 | 305 | |
| 1,1,2-Trichloroethane | 52 | 150 | 25.9 |
| Dibromochloromethane | 53 | 149 | 27.1 |
| Bromoform | 45 | 169 | 28.8 |
| cis-1,3-Dichloropropene | 0 | 227 | 28.7 |
| trans-1,3-Dichloropropene | 17 | 183 | 27.9 |
| Toluene | 47 | 150 | 25.4 |
| Tetrachloroethene | 64 | 148 | 29.7 |
| Chlorobenzene | 37 | 160 | 23 |
| Ethylbenzene | 37 | 162 | 25.2 |
| 1,1,2,2-Tetrachloroethane | 46 | 157 | 22.3 |
| 1,3-Dichlorobenzene | 59 | 156 | 18.2 |
| 1,4-Dichlorobenzene | 18 | 190 | 17.1 |
| 1,2-Dichlorobenzene | 18 | 190 | 17.8 |

## Method 8260 LCS Recovery Limits

| | Water | | Solids | |
|---|---|---|---|---|
| Analyte | Low | High | Low | High |
| Chloromethane | 5.7 | 164 | 66.1 | 115 |
| Vinyl chloride | 37.2 | 152 | 72.2 | 131 |
| Bromomethane | 0 | 287 | 10 | 324 |
| Chloroethane | 0 | 347 | 82.6 | 129 |
| Trichlorofluoromethane | 61.4 | 154 | 82.8 | 148 |
| 1,1-Dichloroethene | 56.6 | 124 | 72.8 | 126 |
| Methylene chloride | 50.7 | 136 | 69.8 | 116 |
| trans-1,2-Dichloroethene | 59.9 | 133 | 80.4 | 118 |
| 1,1-Dichloroethane | 64.3 | 124 | 86.3 | 110 |
| Chloroform | 75.8 | 116 | 85.3 | 114 |
| 1,2-Dichloroethane | 73.3 | 126 | 70.7 | 139 |
| 1,1,1-Trichloroethane | 72.1 | 129 | 86.2 | 124 |
| Benzene | 74.1 | 119 | 85.8 | 120 |
| Carbon tetrachloride | 56.6 | 142 | 78.2 | 126 |
| 1,2-Dichloropropane | 75.4 | 121 | 78.9 | 123 |
| Trichloroethene | 70.7 | 127 | 82.6 | 116 |
| Bromodichloromethane | 71.9 | 122 | 79.5 | 124 |
| 1,1,2-Trichloroethane | 75.6 | 123 | 72 | 137 |
| Dibromochloromethane | 68.1 | 130 | 86.7 | 106 |
| Bromoform | 54.9 | 138 | 69.5 | 133 |
| cis-1,3-Dichloropropene | 74.2 | 128 | 76.4 | 121 |
| trans-1,3-Dichloropropene | 75.8 | 127 | 74.2 | 126 |
| Toluene | 79.6 | 122 | 80.1 | 126 |
| Tetrachloroethene | 71.7 | 139 | 80 | 128 |
| Chlorobenzene | 81.6 | 122 | 83.9 | 123 |
| Ethylbenzene | 71.2 | 140 | 83.8 | 129 |
| 1,1,2,2-Tetrachloroethane | 54.8 | 144 | 77.1 | 145 |
| 1,3-Dichlorobenzene | 73.8 | 113 | 82.7 | 119 |
| 1,4-Dichlorobenzene | 78.9 | 119 | 84 | 119 |
| 1,2-Dichlorobenzene | 79.6 | 121 | 83.3 | 125 |

## Method 8260 MS/MSD Recovery Limits

| | Water | | | Solid | | |
|---|---|---|---|---|---|---|
| Analyte | Low | High | RPD | Low | High | RPD |
| Chloromethane | 10.8 | 155 | 90.9 | 22.5 | 155 | 73 |
| Vinyl chloride | 33 | 151 | 31.2 | 21.5 | 174 | 49.3 |
| Bromomethane | 0 | 243 | 52.8 | 0 | 277 | 112 |
| Chloroethane | 0 | 357 | 29.3 | 0 | 439 | 45 |
| Trichlorofluoromethane | 58.1 | 156 | 81.6 | 52.6 | 145 | 119 |
| 1,1-Dichloroethene | 50.1 | 125 | 31 | 40.5 | 140 | 36.4 |
| Methylene chloride | 55.4 | 130 | 28.7 | 29.8 | 177 | 38.8 |
| trans-1,2-Dichloroethene | 56.9 | 132 | 66.1 | 48.7 | 145 | 36.5 |
| 1,1-Dichloroethane | 68 | 119 | 14.2 | 60.5 | 134 | 24.1 |
| Chloroform | 71.2 | 120 | 25 | 59.8 | 126 | 22.1 |
| 1,2-Dichloroethane | 67.7 | 133 | 23.3 | 55.9 | 149 | 41.2 |
| 1,1,1-Trichloroethane | 63 | 136 | 29 | 51.5 | 149 | 24.4 |
| Benzene | 60.6 | 130 | 23.7 | 54.3 | 134 | 32 |
| Carbon tetrachloride | 47.2 | 149 | 31.1 | 38.2 | 149 | 38.2 |
| 1,2-Dichloropropane | 71.4 | 125 | 26 | 61.2 | 132 | 22.1 |
| Trichloroethene | 48.6 | 139 | 26.9 | 41.8 | 145 | 35.3 |
| Bromodichloromethane | 65.8 | 126 | 28.4 | 53.8 | 134 | 22.4 |
| 1,1,2-Trichloroethane | 40.6 | 165 | 25.9 | 49.2 | 147 | 34.4 |
| Dibromochloromethane | 60.1 | 136 | 27.1 | 47.7 | 137 | 28.4 |
| Bromoform | 40.5 | 146 | 28.8 | 33 | 131 | 41.3 |
| cis-1,3-Dichloropropene | 66.9 | 134 | 28.7 | 31.1 | 176 | 39.2 |
| trans-1,3-Dichloropropene | 69.9 | 130 | 27.9 | 35.6 | 155 | 36.4 |
| Toluene | 69.7 | 130 | 25.4 | 45.3 | 147 | 44.7 |
| Tetrachloroethene | 60.1 | 144 | 29.7 | 51.8 | 139 | 29.1 |
| Chlorobenzene | 75.6 | 127 | 23 | 63.4 | 130 | 19 |
| Ethylbenzene | 60.6 | 144 | 25.2 | 33 | 161 | 70.9 |
| 1,1,2,2-Tetrachloroethane | 46.8 | 168 | 22.3 | 24.9 | 201 | 85.2 |
| 1,3-Dichlorobenzene | 69.3 | 116 | 18.2 | 55.7 | 128 | 89.2 |
| 1,4-Dichlorobenzene | 73.9 | 122 | 17.1 | 61.2 | 133 | 89 |
| 1,2-Dichlorobenzene | 76.7 | 123 | 17.8 | 61.5 | 136 | 86.7 |

| Initial Demonstration of Capability - July 2001 (Spiked conc. = 50 ug/l) | | | | |
|---|---|---|---|---|
| Analyte | Std. Dev. | Std. Dev. Limits | %R | %R Limits |
| Benzene | 1.02 | 6.9 | 87.2 | 70-130 |
| Bromodichloromethane | 0.78 | 6.4 | 93.8 | 70-130 |
| Bromoform | 2.5 | 5.4 | 86.2 | 70-130 |
| Bromomethane | 8.8 | 17.9 | 80.4 | 70-130 |
| 2-Butanone | 1.9 | 4.6 | 91.4 | 70-130 |
| Carbon tetrachloride | 2.5 | 5.2 | 75.8 | 70-130 |
| Chlorobenzene | 2.0 | 6.3 | 104 | 70-130 |
| Chloroethane | 6.5 | 11.4 | 178 | 70-130 |
| Chloroform | 0.72 | 6.1 | 89.5 | 70-130 |
| Chloromethane | 0.74 | 19.8 | 85.8 | 70-130 |
| Dibromochloromethane | 3.0 | 6.1 | 96.5 | 70-130 |
| 1,1-Dichloroethane | 0.82 | 5.1 | 125 | 70-130 |
| 1,2-Dichloroethane | 0.70 | 6.0 | 89.4 | 70-130 |
| 1,1-Dichloroethene | 1.6 | 9.1 | 67.4 | 70-130 |
| cis-1,2-Dichloroethene | 0.97 | NA | 98.1 | 70-130 |
| trans-1,2-Dichloroethene | 1.8 | 5.7 | 86.6 | 70-130 |
| 1,2-Dichloropropane | 0.81 | 13.8 | 97.6 | 70-130 |
| cis-1,3-Dichloropropene | 1.4 | 15.8 | 92.3 | 70-130 |
| trans-1,3-Dichloropropene | 2.0 | 10.4 | 93.4 | 70-130 |
| Ethylbenzene | 2.2 | 7.5 | 98.7 | 70-130 |
| Methylene chloride | 1.4 | 7.4 | 76.8 | 70-130 |
| Styrene | 2.0 | NA | 102 | 70-130 |
| 1,1,1,2-Tetrachloroethane | 2.6 | NA | 102 | 70-130 |
| 1,1,2,2-Tetrachloroethane | 0.86 | NA | 71.1 | 70-130 |
| Tetrachloroethene | 1.4 | 5.0 | 89.4 | 70-130 |
| Toluene | 1.6 | 4.8 | 99.3 | 70-130 |
| 1,1,1-Trichloroethane | 1.7 | 4.6 | 82.6 | 70-130 |
| 1,1,2-Trichloroethane | 1.4 | 5.5 | 89.5 | 70-130 |
| Trichloroethene | 0.36 | 6.6 | 98.3 | 70-130 |
| Trichlorofluoromethane | 3.0 | 10.1 | 73.4 | 70-130 |
| Vinyl chloride | 1.8 | 20 | 73.2 | 70-130 |
| m,p-Xylene | 4.4 | NA | 97.5 | 70-130 |
| o-Xylene | 2.4 | NA | 99.4 | 70-130 |
| 1,1-Dichloropropene | 1.5 | NA | 81.1 | 70-130 |
| 1,2,4-Trichlorobenzene | 1.8 | NA | 93.6 | 70-130 |
| 1,2,4-Trimethylbenzene | 1.5 | NA | 91.8 | 70-130 |
| 1,2-Dibromoethane | 2.8 | NA | 95.7 | 70-130 |
| 1,2-Dichlorobenzene | 1.8 | 7.1 | 98.3 | 70-130 |
| 1,3,5-Trimethylbenzene | 2.1 | NA | 95.5 | 70-130 |
| 1,3-Dichlorobenzene | 1.7 | 5.5 | 89.3 | 70-130 |
| 1,3-Dichloropropane | 1.4 | NA | 99.9 | 70-130 |
| 1,4-Dichlorobenzene | 2.4 | 7.1 | 96.6 | 70-130 |
| 2-Chlorotoluene | 3.5 | NA | 104 | 70-130 |
| 4-Chlorotoluene | 2.3 | NA | 101 | 70-130 |
| Bromobenzene | 3.1 | NA | 102 | 70-130 |
| Dibromomethane | 0.81 | NA | 87.3 | 70-130 |
| Dichlorodifluoromethane | 1.3 | NA | 67.0 | 70-130 |
| Isopropylbenzene | 1.9 | NA | 94.7 | 70-130 |
| n-Butylbenzene | 1.4 | NA | 89.0 | 70-130 |
| n-Propylbenzene | 2.2 | NA | 93.4 | 70-130 |
| p-Isopropyltoluene | 0.98 | NA | 91.3 | 70-130 |
| sec-Butylbenzene | 1.3 | NA | 85.8 | 70-130 |
| tert-Butylbenzene | 0.98 | NA | 91.0 | 70-130 |

# STANDARD OPERATING PROCEDURE FOR SEMI-VOLATILE ORGANIC COMPOUNDS (SVOA) BY EPA METHOD 625 AND SW-846 METHOD 8270C

Originating Author: Nancy Tavitas
Revision Author: Jeffrey M. Loewe

This SOP is effective upon signed approval by the following:

Nancy L Tavitas — Unit Supervisor — 11/13/01 — Date

Jeffrey M. Loewe — QA/QC Director — 11-13-2001 — Date

**DISCLAIMER: This SOP has been developed for use at the SIMALABS International, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.**

# 1.0 TABLE OF CONTENTS

## 2.0 SCOPE AND APPLICATION

2.1 This is a GC/MS procedure for the determination of semi-volatile organic compounds. This procedure is applicable to the analysis of extracts from aqueous, non-aqueous liquid, and solid matrix samples. The routine reporting limits are listed in the table below. These limits may vary due to matrix effects.

| ANALYTE | PQL, ug/l | PQL, ug/kg | ANALYTE | PQL, ug/l | PQL, ug/kg |
|---|---|---|---|---|---|
| Pyridine | 10 | 330 | 2,6-dinitrotoluene | 10 | 330 |
| N-Nitrosodimethylamine | 10 | 330 | 3-nitroaniline | 50 | 1660 |
| Phenol | 10 | 330 | Acenaphthene | 10 | 330 |
| Aniline | 10 | 330 | 2,4-dinitrophenol | 20 | 660 |
| bis (2-chloroethyl) ether | 10 | 330 | Dibenzofuran | 10 | 330 |
| 2-chlorophenol | 10 | 330 | 4-nitrophenol | 50 | 1660 |
| 1,3-dichlorobenzene | 10 | 330 | 2,4-dinitrotoluene | 10 | 330 |
| 1,4-dichlorobenzene | 10 | 330 | Fluorene | 10 | 330 |
| 1,2-dichlorobenzene | 10 | 330 | 4-chlorophenyl phenyl ether | 10 | 330 |
| Benzyl alcohol | 10 | 330 | Diethyl phthalate | 10 | 330 |
| bis (2-chloroisopropyl) ether | 10 | 330 | 4-nitroaniline | 50 | 1660 |
| 2-methylphenol | 10 | 330 | 4,6-dinitro-2-methylphenol | 50 | 1660 |
| Hexachloroethane | 10 | 330 | N-Nitrosodiphenylamine | 10 | 330 |
| N-nitrosodi-n-propylamine | 10 | 330 | 1,2-diphenylhydrazine | 10 | 330 |
| 3/4-methylphenol | 10 | 330 | 4-bromophenyl phenyl ether | 10 | 330 |
| Nitrobenzene | 10 | 330 | Hexachlorobenzene | 10 | 330 |
| Acetophenone | 10 | 330 | Pentachlorophenol | 50 | 1660 |
| Isophorone | 10 | 330 | Phenanthrene | 10 | 330 |
| 2-nitrophenol | 10 | 330 | Anthracene | 10 | 330 |
| 2,4-dimethylphenol | 10 | 330 | Carbazole | 10 | 330 |
| bis (2-chloroethoxy) methane | 10 | 330 | Di-n-butyl phthalate | 10 | 330 |
| 2,4-dichlorophenol | 10 | 330 | Fluoranthene | 10 | 330 |
| Benzoic acid | 50 | 1600 | Benzidine | 10 | 330 |
| 1,2,4-trichlorobenzene | 10 | 330 | Pyrene | 10 | 330 |
| Naphthalene | 10 | 330 | Butyl benzyl phthalate | 10 | 330 |
| 2,6-dichlorophenol | 10 | 330 | Benzo(a)anthracene | 10 | 330 |
| 4-chloroaniline | 20 | 660 | 3,3'-dichlorobenzidine | 50 | 1660 |
| Hexachlorobutadiene | 10 | 330 | Chrysene | 10 | 330 |
| 4-chloro-3-methylphenol | 20 | 660 | bis (2-ethylhexyl) phthalate | 10 | 330 |
| 2-methylnaphthalene | 10 | 330 | Di-n-octyl phthalate | 10 | 330 |
| Hexachlorocyclopentadiene | 10 | 330 | Benzo(b)fluoranthene | 10 | 330 |
| 2,4,5-trichlorophenol | 10 | 330 | Benzo(k)fluoranthene | 10 | 330 |
| 2,4,6-trichlorophenol | 10 | 330 | Benzo(a)pyrene | 10 | 330 |
| 2-chloronaphthalene | 10 | 330 | Indeno(1,2,3-cd)pyrene | 10 | 330 |
| 2-nitroaniline | 50 | 1660 | Dibenz(a,h)anthracene | 10 | 330 |
| Acenaphthylene | 10 | 330 | Benzo(g,h,i)perylene | 10 | 330 |
| Dimethyl phthalate | 10 | 330 | | | |

2.2 Methods 8270C and 625 can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and are capable of being eluted, without derivitization, as sharp peaks from a gas chromatographic fused silica capillary column. Some of these compounds are: polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, nitrosamines, ethers, anilines, aromatic nitro compounds, and phenols.

## 3.0 SUMMARY

3.1 Semi-volatile organic compounds are extracted from the sample matrix using methylene chloride. After concentration, the extract is injected into the GC/MS system and analyzed following the more stringent criteria between SW-846 Method 8270C and EPA Method 625.

## 4.0 DEFINITIONS

4.1 Accuracy – The degree of agreement of a measured value with the true or expected value of the quantity of concern (% recovery of a known spiked analyte).

4.2 Aliquot – A measured portion of a sample, or solution, taken for sample preparation or analysis.

4.3 Analyte – The specific component measured in a chemical analysis.

4.4 Analytical Batch – A group of samples which are analyzed, at the instrument level, together using the same method, reagents and apparatus within the same time period. Typically, these are samples in the same workgroup (WG) in the analytical department of the LIMS.

4.5 Blank – An artificial sample designed to assess specific sources of laboratory contamination. There are several types of blanks, which monitor a variety of processes:

- Calibration Blank – An aliquot of the standard diluent (water or organic solvent) that is not carried through the sample preparation scheme. It is analyzed to verify that the analytical system is free from contamination. Also referred to as an instrument blank or solvent blank.
- Field Blank – blanks that are collected in the field and analyzed to determine the level of contamination introduced into the sample due to sampling technique.
- Method Blank – An aliquot of lab pure water or solid matrix taken through sample preparation (when required) and analysis. It is a test for contamination in sample preparation and analyses. Also referred to as a Preparation Blank.

4.6 Bias – The deviation of a measured value from a known or accepted value due to matrix effects or method performance. Bias may be determined quantitatively to correct measured values. Bias may be positive or negative.

4.7 Calibration – The establishment of an analytical curve based on the absorbance, response, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type and concentration of acids, solvents, or other solutions used in the sample preparation.

4.8 Calibration Factor – A measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the volatile and semi-volatile fractions.

4.9 Continuing Calibration Verification Standard (CCV) – A calibration standard used to verify the continued acceptability of the initial calibration curve. A CCV must be analyzed at the beginning of each analytical sequence following an acceptable instrument tune.

4.10 Detection Limit – The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

- MDL – Method detection limit. The minimum concentration of a substance that can be measured and reported with a 99% degree of confidence. MDLs are determined by analyzing a minimum of seven consecutive standards that have been processed through all preparatory steps. These standards must meet criteria of bias and precision.

- PQL – The Practical Quantitation Limit is the lowest concentration that can reliably be achieved within specified limits of precision and accuracy during routine laboratory operating conditions. Typically, the PQL is a value in the range of 5 - 10 times the MDL. Also referred to as the Estimated Quantitation Limit (EQL).

4.11 Initial Calibration Verification (ICV) – A standard used to verify the accuracy of calibration standards. Prepared from a second source than that of the calibration standards, and analyzed immediately after the generation of a new calibration curve, its known value is measured against the calibration curve. This determines the integrity of working standards. Also referred to as an external verification standard or check standard.

4.12 Holding Time – The maximum storage time allowed between sample collection, sample extraction, and sample analysis.

4.13 Internal Standard (I.S.) – Applicable to GC, GC/MS and LC analyses only. A compound(s), not of interest as a target compound, which is added to all samples, QC samples, and calibration standards just prior to instrument analysis. Internal standards are used as the basis for quantitation of target compounds for GC and GC/MS analysis.

4.14 Laboratory Control Sample (LCS) – An aliquot of analyte-free water or solid matrix spiked with target analytes or compounds representative of target analytes. The sample is carried through the entire analytical process and analyte recovery is used to monitor method performance. Also referred to as a laboratory fortified blank (LFB).

4.15 Laboratory Control Sample Duplicate (LCSD) – An aliquot of analyte-free water or solid matrix spiked with the identical amount(s) of target analyte(s) as the LCS. Results of the two spikes are used to assess both the bias and precision of a

method with a given sample matrix. Also referred to as a laboratory fortified blank duplicate (LFB DUP).

4.16 Matrix – The component or substrate which may contain the analyte of interest. Matrices are limited to the following: aqueous (includes extracts from the TCLP or other extraction procedure, groundwater, surface water, and wastewater), drinking water (potable water and laboratory pure water), non-aqueous liquid (organic liquid having <15% settleable solids), and solid (includes sediment, sludge, and soil).

4.17 Matrix Spike (MS) – An aliquot of a sample that is spiked with a known amount of target analyte(s). Recovery of the matrix spike, expressed as percent recovery, is used to assess the bias of a method in a given sample matrix. Also referred to as a laboratory fortified sample matrix (LFSM).

4.18 Matrix Spike Duplicate (MSD) – An aliquot of the same sample used for the MS, spiked with the identical amount(s) of target analyte(s) as the MS. Results of the two spikes are used to assess both the bias and precision of a method with a given sample matrix. Also referred to as a laboratory fortified sample matrix duplicate (LFSM DUP).

4.19 Percent Difference (%D) – Used to compare two values, the percent difference indicates both the direction and the magnitude of the comparison. The percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

$$\%D = \frac{(X - Y)}{X} * 100$$

where: X = value 1
Y = value 2

4.20 Percent Recovery – A measure of accuracy that is calculated as the measured value relative to the true value, expressed as a percent.

$$\%R = \frac{MV}{TV} * 100$$

where: MV = measured value
TV = true value

4.21 Precision – The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the comparability of results from duplicate or replicate analyses. (%RPD between the recoveries of two known analyte spikes, and %RSD between the recoveries of three or more measurements).

4.22 Preparation Batch – A group of samples of similar composition which are prepared together using the same method, reagents and apparatus within a 24 hour calendar day or every 20 samples, whichever is more frequent.

4.23 Preservative – A reagent added to a sample, or an action used, to prevent or slow decomposition or degradation of a target analyte or a physical process. Thermal and chemical preservation may be used in tandem to prevent sample deterioration.

4.24 Relative Percent Difference (% RPD) – Used to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

$$\% \text{ RPD} = \frac{|X - Y|}{(X + Y) / 2} * 100$$

where: X = value 1
Y = value 2

4.25 Relative Response Factor – Applicable to GC, GC/MS, and LC analyses only. A measure of the relative response of an analyte compared to that of its internal standard. Relative response factors (RRF) are determined by analysis of calibration standards and are used in the quantitation of target analytes in samples. RRF is calculated as follows:

$$\text{RRF} = \frac{Ax \times Cis}{Ais \times Cx}$$

where: Ax = area of the compound of interest measured
Cis = concentration of the internal standard
Ais – area of the internal standard
Cx = concentration of the analyte of interest

4.26 Relative Retention Time (RRT) – The ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$\text{RRT} = \frac{RT_c}{RT_{is}}$$

where: $RT_c$ = Retention time for the target or surrogate in continuing calibration
$RT_{is}$ = Retention time for the internal standard in calibration standard or sample

4.27 Relative Standard Deviation (% RSD) – Used to compare more than two values, the relative standard deviation is based on the variance and the mean of the values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

$$\% \text{ RSD} = \frac{s}{\text{avg.}} * 100$$

where: s = standard deviation
avg. = arithmetic average

4.28 Retention Time – The time elapsed from sample injection until the specific compound elutes or exits the chromatographic column at the detector. Each analyte has a characteristic retention time on a specific column allowing this information is used to qualitatively identify the analytes in the sample.

4.29 Sample – A portion of material supplied by the client for analysis.

4.30 Surrogate Compound – Applicable to GC, GC/MS, and LC analyses only. Compound that behaves similarly, with respect to the analytical method, as the analytes of interest but is not normally found in environmental samples. Often, surrogates are isotopic homologues of target analytes. Surrogate(s) are added to all blanks, samples and QC samples prior to preparation and analysis. Recovery of surrogates is used to assess method performance.

4.31 Tune – Analysis of a compound to verify the operating conditions of the instrument by comparing the ion abundance of various masses.

## 5.0 INTERFERENCES

5.1 Interferences are minimized through the use of ion counts and internal standards.

5.2 Contamination by carryover can occur when a low-level sample is analyzed after a high level sample. Solvent blanks should be analyzed in the instances to check for carryover effects.

## 6.0 SAFETY

6.1 Eye protection must be worn at all times while in the laboratory.

6.2 Lab coats and gloves are recommended. Avoid direct contact with reagents, standards, and/or samples.

6.3 Consult the Material Safety Data Sheets (MSDS) for each chemical used for information regarding fire hazard, toxicity, first aid, storage, disposal, spill procedures, and recommended protective equipment.

6.4 Chemicals having the potential to produce toxic fumes must be handled in a fume hood.

## 7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

7.1 All volumetric glassware used shall be ASTM Class A.

7.2 HP5890 Series II Gas Chromatograph with electronic pressure control (EPC)

7.3 Chromatographic column: DB-5MS, length 30m, ID 0.25mm, film thickness 0.5 um (J&W Scientific part #122-5536)

7.4 Mass Selective Detector: HP 5971A

7.5 Autosampler/controller: HP7673

7.6 Ion Gauge Controller: HP 59822B

7.7 Computer with MS Chemstation software, monitor, and printer

7.8 Syringes: various sizes including 10 ul, 500 ul, and 1000 ul

7.9 Autosampler vials: 2 ml size with screw tops as well as 2 ml size with crimp tops

7.10 Crimper tool

7.11 200 ul glass inserts

## 8.0 REAGENTS AND STANDARDS

8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

8.2.1 Methylene chloride, pesticide quality or greater

8.2.2 Methanol, pesticide quality or greater

8.3 Standards

8.3.1 Stock Tune Solution, 1000 ug/ml DFTPP, Benzidine, Pentachlorophenol, and 4,4'-DDT in methylene chloride: Accustandard Cat #M-625-TS-20X

8.3.2 Working Tune Solution, 50 ug/ml each: In a 10 ml volumetric flask, dilute to the mark 250 ul of the Stock Tune Solution with methylene chloride. Transfer the dilution into a screw top vial and store it in the Organics standard freezer.

8.3.3 Stock Internal Standard (I.S.), 4000 ug/ml each in methylene chloride.

| VENDOR | CAT # | COMPOUNDS |
|---|---|---|
| Accustandard | Z-014J-PAK SVOA | 1,4-dichlorobenzene-d4; naphthalene-d8; acenaphthalene-d10; phenanthrene-d10; chrysene-d12; perylene-d14 |

8.3.4 Stock Acid Surrogate Standard, 10000 ug/ml each in methanol.

| VENDOR | CAT # | COMPOUNDS |
|---|---|---|
| Supelco | 4-7261 | 2-fluorophenol; phenol-d5; 2,4,6-tribromophenol |

8.3.5 Stock Base/Neutral Surrogate Standard, 5000 ug/ml each in methanol.

| VENDOR | CAT # | COMPOUNDS |
|---|---|---|
| Supelco | 4-7262 | Nitrobenzene-d5; 2-fluorobiphenyl; terphenyl-d14 |

8.3.6 Stock Calibration Standards, 2000 ug/ml each in methylene chloride.

| MIX # | VENDOR | CAT # | COMPOUNDS |
|---|---|---|---|
| SVOA 1 | Accustandard | M-8270-01 | See Table in Section 18.0 |
| SVOA 2 | Accustandard | M-8270-02 | |
| SVOA 3 | Accustandard | M-8270-03 | |
| SVOA 4A | Accustandard | M-8270-04A | |
| SVOA 4B | Accustandard | M-8270-04B | |
| SVOA 5 | Accustandard | M-8270-05 | |
| SVOA6 | Accustandard | M-8270-06 | |
| | Supelco | 4-8076 | Carbazole |
| | Supelco | 4-8305-U | Pyridine |

8.3.7 Intermediate Calibration Standards, 200 ug/ml each: In a 5 ml volumetric flask, dilute 500 ul of the Stock Calibration Standards, 100 ul of the Stock Acid Surrogate Standard, and 200 ul of the Stock Base/Neutral Surrogate Standard to volume with methylene chloride. Store in the Organics standard freezer.

8.3.8 Working Calibration Standards: In separate 1 ml volumetric flasks, prepare the following dilutions with methylene chloride. Transfer the dilutions into separate 1 ml screw top vials and store them in the Organics standard freezer. The I.S. concentration is 40 ug/ml.

| Linearity Standard # | Vol. Stock Cal. Std, ul | Vol. Stock I.S., ul | Final Conc. ug/ml |
|---|---|---|---|
| 1 | 100 | 10 | 20 |
| 2* | 250 | 10 | 50 |
| 3** | 400 | 10 | 80 |
| 4 | 600 | 10 | 120 |
| 5 | 800 | 10 | 160 |

* typically used as the CCV
** used as CCV for NC compliance samples

8.3.9 Stock ICV Standards:

| VENDOR | CAT # | COMPOUNDS | CONC., ug/ml |
|---|---|---|---|
| Supelco | 506508 | See Table in Section 18 | 1000 |
| Accustandard | S-9076 (custom mix) | Benzoic Acid; Benzyl alcohol; 2,6-Dichlorophenol; N-Nitrosophenylamine; Benzidine; 3,3'-Dichlorobenzidine; Acetophenone; Pyridine; Aniline | 1000 |

8.3.10 Working ICV Standard, 50 ug/ml each: In a 1 ml volumetric flask, dilute 50 ul of the stock ICV standards, 10 ul of the stock Base/Neutral surrogate standard, 5 ul of the stock Acid surrogate standard, and 10 ul of the stock internal to the mark with methylene chloride. Transfer the dilution into a screw top vial and store it in the Organics standard freezer. The final concentration of the surrogates is 50 ug/ml each and the concentration of the internal standards is 40 ug/ml each.

8.3.11 LCS/MS/MSD: When prepared as detailed in the preparation SOPs, the final concentrations are 100 ug/l (3333 ug/kg) for all spiked analytes.

## 9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.

9.2 Extracts must be stored in the range of 0.1-6°C (0.1-4°C for North Carolina compliance monitoring). Extracts are stored in the Organics sample cooler located in the GC/SVOA lab.

9.3 Analysis must be performed within 40 days of extraction.

## 10.0 QUALITY CONTROL

10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Analyze four separate standards prepared in the range of 8-10 times the method detection limit listed in section 14.0. These standards must be from a source different from that used for calibration and taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.2 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Analyze a minimum of seven (maximum of ten) standards prepared in the range of 2-5 times the method detection limit listed in section 14.0 or an estimated detection limit. These standards must be taken through the entire analytical procedure. Submit the data to the QA department for evaluation. Refer to the Capability and Detection Limit Studies SOP for details.

10.3 A *DFTPP Tune Check* must be performed at the beginning of each 12-hour sequence. A 1 ul (50 ng) injection of the working tune solution (which contains DFTPP) must meet the following ion abundance criteria. If the criteria are not met, reanalyze. An acceptable tune must be obtained before samples can be analyzed.

DFTPP (DECAFLUOROTRIPHENYLPHOSPHINE)
KEY IONS and ION ABUNDANCE CRITERIA
METHODS 625 / 8270C

| Mass | Ion Abundance Criteria |
|---|---|
| | |
| 51 | 30 – 60% of mass 198 |
| | |
| 68 | < 2% of mass 69 |
| 69 | Present |
| 70 | < 2% of mass 69 |
| | |
| 127 | 40 – 60% of mass 198 |
| | |
| 197 | < 1% of mass 198 |
| 198 | Base peak, 100% relative abundance |
| 199 | 5 – 9% of mass 198 |
| | |
| 275 | 10 – 30% of mass 198 |
| | |
| 365 | > 1% of mass 198 |
| | |
| 441 | Present but less than mass 443 |
| 442 | > 40% of mass 198 |
| 443 | 17 – 23% of mass 442 |

10.4 The *Tailing Factors* for Benzidine and Pentachlorophenol must be evaluated at the beginning of each 12-hour sequence. A 1 ul injection of the working tune solution (which contains Benzidine and Pentachlorophenol) must yield a tailing factor less than 3.0 for Benzidine and less than 5 for Pentachlorophenol. The tailing factors

are calculated (according to Figure 13 in Method 625) by the instrument software and documented on the instrument printout. If the tailing factor criterion cannot be achieved, perform instrument maintenance or re-prepare the tune solution, and reanalyze. Instrument maintenance may include cutting off the first 6-12 inches of the column. Tailing factor criteria must be met before samples can be analyzed.

10.5 *Internal Standards* (I.S.) must be added to all standards, QC samples and environmental samples. Acceptance criteria for CCV are RT ± 30 seconds from that in the midpoint level standard of the most recent initial calibration and area counts within the range of -50 to +100% of those in the same linearity standard. These criteria are evaluated by the data system and printed on the Continuing Calibration Report. Failures are automatically flagged on this report. If acceptance criteria are not met for the CCV, perform instrument maintenance or perform a new initial calibration. As there are no absolute requirements for I.S. recovery in samples, the acceptance criteria for the CCV should be used as guidance in evaluating the response for samples. If the sample internal standard response does not meet the CCV acceptance criteria, evaluate the chromatogram for obvious matrix effects/interferences. If interferences are evident, the sample should be diluted and reanalyzed. If the internal standard response for the sample is below 25% the analyst should consult with the Unit Supervisor and use discretion on whether to report or re-extract and analyze the sample. This discretion is dependent on the experience of the analyst and their Supervisor and factors such as a consistent response for the I.S. should be considered. If reanalysis yields poor internal standard response, or if reanalysis is performed beyond the hold time, both sets of data should be reported to the client and a Case Narrative written.

10.6 *Surrogate* (SURR) compounds must be added to all quality control samples, blanks, and samples. Acceptance criteria are the statistically generated matrix-specific recovery limits listed below. These criteria are evaluated by the LIMS and printed on the LIMS QC Report and analytical report submitted to the client. One acid and one Base/Neutral surrogate may be outside of the acceptance criteria. Failures are automatically flagged with a "S" on these reports. If more than one of each fraction surrogate is out, re-extract and analyze. If reanalysis does not yield acceptable recovery, both sample sets must be supplied to the client. If reanalysis is performed beyond the maximum allowable hold time, both sample sets must be supplied to the client and the appropriate result flagged with a "H" qualifier as defined in the LIMS. If insufficient sample is available for re-extraction the original result should be reported and qualified in the Case Narrative to state this as the reason for no re-extraction and reanalysis being performed. If the acceptance criteria are not met for a method blank but are met for the QC samples and environmental samples, report the sample results qualified for the MB failure in the Case Narrative.

| Surrogate Recovery Limits | | | | |
|---|---|---|---|---|
| | Water | | Solid | |
| Analyte | Low | High | Low | High |
| 2,4,6-Tribromophenol | 10 | 120 | 10 | 107 |
| 2-Fluorobiphenyl | 10 | 109 | 10 | 124 |
| 2-Fluorophenol | 10 | 84.7 | 10 | 91.4 |
| Nitrobenzene-d5 | 10 | 121 | 10 | 139 |
| Phenol-d5 | 10 | 100 | 10 | 97.5 |
| Terphenyl-d14 | 10 | 130 | 10 | 157 |

10.7 An *Initial Calibration Verification* (ICV) standard must be immediately after the initial linearity (ICAL). This is the analysis of a second source standard. Acceptance criteria are the statistically generated recovery limits listed in section 18.0. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, analysis must be stopped, the problem corrected, and the instrument recalibrated.

10.8 A *Continuing Calibration Verification* (CCV) standard is a calibration source standard and must be analyzed at the beginning of each analytical sequence following an acceptable instrument tune. The 12-hour analytical sequence begins with the injection of DFTPP, continues through the analysis of the CCV, samples and QC samples. Acceptance criteria for Method 8270 are RF for SPCCs $\geq$ 0.050 and RF for CCCs $\leq$ 20% difference from the initial calibration. Acceptance criteria for Method 625 are responses $\leq$ 20% difference from the initial calibration. If acceptable criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, recalibrate. Samples with a non-detect concentration may be reported if the CCV fails to meet the acceptance criteria with a positive bias.

| SPCCs | |
|---|---|
| N-nitroso-di-n-propylamine | 2,4-dinitrophenol |
| Hexachlorocyclopentadiene | 4-nitrophenol |
| | |

| CCCs | |
|---|---|
| *B/N Fraction* | *Acid Fraction* |
| Acenaphthene | 4-Chloro-3-methylphenol |
| 1,4-dichlorobenzene | 2,4-dichlorophenol |
| Hexachlorobutadiene | 2-nitrophenol |
| Diphenylamine | Phenol |
| Di-n-octyl phthalate | Pentachlorophenol |
| Fluoranthene | 2,4,6-Trichlorophenol |
| Benzo(a)pyrene | |

10.9 A *Method Blank* (MB) must be extracted and analyzed with each preparation batch of up to 20 samples per day (at a minimum of 1 per day) per matrix. Acceptance criteria are no detects above the PQL, however, the MB remains acceptable if the blank concentration is less than 1/10 of the sample concentration, or if there were no detects in the sample. If the acceptance criteria are not met, re-extract the blank and the affected samples if sufficient sample is available. If insufficient sample is available for reanalysis the original result should be reported and qualified in the Case Narrative. Samples associated with a contaminated blank must be reported with a "B" qualifier as defined in the LIMS. If sample reanalysis

was performed beyond the maximum allowable hold time, both sets of results must be supplied to the client and flagged with a "H", as appropriate, to note the hold time exceedance. When a MB is contaminated, corrective action steps must be taken to identify and eliminate the cause of the contamination.

10.10 A *Laboratory Control Sample* (LCS) must be extracted and analyzed with each preparation batch of up to 20 samples per day (at a minimum of 1 per day) per matrix. Acceptance criteria are the statistically generated recovery limits listed in section 18.0 for Method 8270C or the recovery limits in Table 6 of Method 625, as appropriate. However, the LCS remains acceptable if the failed recovery is positive bias (high) and there are no detects in the sample. If the acceptance criteria are not met, reanalyze or re-extract as appropriate. If this reanalysis does not meet the acceptance criteria, the affected samples from that batch must be re-extracted and analyzed. If insufficient sample is available for reanalysis the original result should be reported and qualified in the Case Narrative. If the hold time has expired and reanalysis performed, both sets of data should be reported and the appropriate result flagged with a "H" qualifier as defined in the LIMS. When prepared for samples being analyzed by Method 8270C, the spiked analytes consist of the compounds listed in section 5.5.1 of Method 3500B. When prepared for samples being analyzed by Method 625, the spike analytes consist of the compounds listed in section 5.5.1 of Method 3500B plus an additional 10-12 compounds listed in Table 6 of Method 625. These additional compounds are changed at approximate 6 month intervals so that all of the Table 6 compounds can be evaluated over a two year period.

10.11 A *Matrix Spike and Matrix Spike Duplicate* (MS/MSD) must be extracted and analyzed with each preparation batch of up to 20 samples per day (at a minimum of 1 per day). When insufficient sample is available for a MSD, a duplicate laboratory control sample (LCSD) should be extracted and analyzed. Acceptance criteria are the statistically generated recovery limits listed in section 18.0 for Method 8270C or the recovery limits in Table 6 of Method 625, as appropriate, however, the LCS remains acceptable if the failed recovery is positive bias (high) and there are no detects in the sample. If the acceptance criteria are met in the MSD, continue. If the accuracy criteria are not met in the MS or MSD, and the LCS is in control, assume matrix interference and report the results with a "S" qualifier as defined in the LIMS. Precision criteria are the statistically generated limits listed in section 18.0. If the precision criteria are not met, report the results with a "R" qualifier as defined in the LIMS. The list of spiked compounds is the same as that for the LCS.

## 11.0 CALIBRATION AND STANDARDIZATION

11.1 Perform the required preventative maintenance as necessary. A new Initial Calibration (linearity) is required as indicated by the quality control elements. The concentration of the low standard must be set near or below the routine PQL for each compound.

11.2 Instrument conditions are as follows:

Zone Temperatures

| | | |
|---|---|---|
| Inlet A: | On | Setpoint: 250°C |
| Inlet B: | Off | |
| Detector A: | Off | |
| Detector B: | On | Setpoint: 280°C |

DFTPP.m

| | |
|---|---|
| Injection size: | 1 ul |
| Initial Temperature: | 150°C |
| Initial Time: | 0.75 minute |
| Rate: | 25°C / minute |
| Final Temperature: | 320°C |
| Final Time: | 1.25 minutes |
| Rate: | 12°C / minute |
| Final Temperature: | 250°C |
| Final Time: | 0.0 minutes |
| Run Time: | 8.8 minutes |

8270-Aq.m

| | |
|---|---|
| Injection size: | 1 ul |
| Initial Temperature: | 40°C |
| Initial Time: | 5.50 minute |
| Rate A: | 12°C / minute |
| Final Temperature: | 250°C |
| Final Time: | 0.0 minutes |
| Rate B: | 20°C / minute |
| Final Temperature: | 320°C |
| Final Time: | 8.5 minutes |
| Run Time: | 35.0 minutes |

11.3 Set up analysis sequence in Sample Table Log in *Sequence* in main menu.

11.4 Enter the sequence as it is to be run, including the applicable analysis method. An ICV standard must be analyzed following the generation of a new linearity and the acceptance criteria met before continuing with sample analysis. If the acceptance criteria are met, rename the method "SVOCmmdd", where mmdd designates the month and date of the new linearity.

11.5 Following the DFTPP tune, the daily calibration standard (CCV) must be run.

11.6 Click on *Position and Run.*

11.7 After each sample has run, the report is retrieved and quantitated against the current initial linearity. The chromatogram is examined to determine that all compounds present have been detected.

11.8 For standards, the software will calculate RF values and % difference values for all analytes. The average RF for the SPCCs must be $\geq$ 0.050, and the %RSD for the CCCs must be < 30%. Where these criteria are met and the RSD of all individual

compounds is ≤15%, the calibration is considered linear and the average RF is used for concentration calculations.

11.8.1 If the RSD > 15, averaging may be used to identify a linear curve. This technique assesses the average %RSD of all compounds in the calibration curve. If the RSD of all (target and non-target) compounds is ≤ 15%, the calibration can be considered acceptable and the average RF used. When used, the fact of its use and average RSD must be reported to the data user.

11.9 If the linearity requirements are not met, take appropriate corrective actions and recalibrate. Analysis of environmental samples cannot proceed without the generation of an acceptable linearity.

## 12.0 PROCEDURE

12.1 Once it has been determined that the tune and calibration verification meet their respective criteria, the analysis of samples can begin. A typical sequence follows the order: Tune, CCV, samples, and QC samples.

12.1.1 To prepare the extracts for analysis, withdraw 200 ul of the extract with a syringe and place it into a 1.0 ml autosampler vial containing a glass insert. Add 2.0 ul of the stock internal standard. The concentration of the internal standards will be 40 ug/ml each. NOTE: All extracts, including dilutions, must be spiked with the internal standards at 40 ug/ml.

12.2 If the concentration of any target compound in a sample exceeds the initial calibration range, a new aliquot of that extract must be diluted and analyzed.

## 13.0 CALCULATIONS AND DATA HANDLING

13.1 After review, enter final results into the LIMS system. Results flagged by the LIMS with an "E" qualifier are above the linear range of the instrument. There is less certainty in these data and, if sufficient sample and holding time are available, should be reanalyzed at an appropriate dilution. Details on the procedure for entering analytical data are in the Data Entry SOP. The peak integrations must be performed according to the Manual Integration of Chromatographic Peaks SOP.

13.2 Calculate the response factor as follows:

$$RF = (A_x)(C_{IS}) / (A_{IS})(C_x)$$

Where: $A_x$ = Area of characteristic ion for compound being measured
$A_{IS}$ = Area of characteristic ion for compound being measured
$C_{IS}$ = Concentration of the specific internal standard
$C_x$ = Concentration of the compound being measured

13.3 The LIMS calculates the sample concentration as follows:

$$\text{Conc.} = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{IS})(RF)(V_o)(V_i)}$$

Where: $A_x$ = Area of characteristic ion for compound being measured
$I_s$ = Amount of internal standard injected (ng)
$V_t$ = Volume of total extract, taking into account dilution
$A_{IS}$ = Area of characteristic ion for the internal standard
RF = Initial average response factor for compound being measured
$V_o$ = Volume of water extracted (L), or mass of soil extracted (kg)
$V_i$ = Volume of extract injected (ul)
DF = dilution factor

13.4 The LIMS calculates the dry-weight concentration for solid samples as follows:

$$\text{Conc. Dry} = \frac{\text{(wet weight conc.)}}{\text{(100 - \% Moisture)}}$$

13.5 The data system software evaluates the retention time and comparison to the characteristic ions to identify any compounds present. The characteristic ions of the reference spectrum are the three ions of greatest intensity (or any ions having a relative intensity greater than 30% if less than three ions are present). The following criteria are used for qualitative identification.

13.5.1 The characteristic ions of a compound must have a relative retention time of ± 0.06 units of the standard (RT ± 30 sec for Method 624).

13.5.2 The relative intensities of the characteristic ions are within 20% of those ions in the reference spectrum (30% for Method 8260).

13.5.3 Structural isomers having a resolution of < 25% are considered isomeric pairs.

## 14.0 METHOD PERFORMANCE

14.1 Initial Demonstration of Capability – Data from a typical IDC study is in section 18.0

## 15.0 POLLUTION PREVENTION

15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

## 16.0 WASTE MANAGEMENT

16.1 Refer to the SIMALABS International Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, extract or standard.

## 17.0 REFERENCES

17.1 USEPA Method 625 as found as an Appendix to 40 CFR 136.

17.2 SW-846 Method 8270C

17.3 SIMALABS International SOP Preparation of Aqueous Samples Using Liquid-Liquid Extraction by SW-846 Method 3510C, current revision.

17.4 SIMALABS International SOP Preparation of Aqueous Samples Using Continuous Liquid-Liquid Extraction by SW-846 Method 3520C, current revision.

17.5 SIMALABS International SOP Preparation of Non-Aqueous Samples Using Sonication by SW-846 Method 3550B, current revision.

17.6 SIMALABS International Quality Assurance Plan, current revision

## 18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

| Calibration Standard Compounds | |
|---|---|
| **M-8270-01** | Aniline Benzyl alcohol Bis(2-Chloroethyl)ether 1,2-Dichlorobenzene Bis(2-Chloroisopropyl)ether 2-Chlorophenol 1,3-Dichlorobenzene 1,4-Dichlorobenzene Hexachloroethane 2-Methylphenol 4-Methylphenol N-Nitrosodimethyamine N-Nitrosodi-n-propylamine Phenol |
| **M-8270-02** | Acetophenone Benzoic acid bis(2-Chloroethoxy)methane 4-Chloroaniline 4-Chloro-3-methylphenol 2,4-Dichlorophenol 2,6-Dichlorophenol 2,4-Dimethylphenol Hexachlorobutadiene Isophorone Naphthalene Nitrobenzene 2-Nitrophenol 1,2,4-Trichlorobenzene 2-Methylnaphthalene |
| **M-8270-03** | Acenaphthene Acenaphthylene 2-Chloronaphthalene 4-Chlorophenyl phenyl ether Dibenzofuran Diethyl phthalate 2,4-Dinitrophenol 2,4-Dinitrotoluene 2,6-Dinitrotoluene Fluorene Hexachlorocyclopentadiene 2-Nitroaniline 3-Nitroaniline 4-Nitroaniline 4-Nitrophenol 2,4,5-Trichlorophenol 2,4,6-Trichlorophenol Dimethylphthalate |
| **M-8270-04A** | Anthracene 4-Bromophenyl phenyl ether Di-n-butyl phthalate 4,6-Dinitro-2-methylphenol Fluoranthene Hexachlorobenzene Pentachlorophenol Phenanthrene |
| **M-8270-04B** | 1,2-Diphenylhydrazine N-Nitrosodiphenylamine |
| **M-8270-05** | Benzidine Benzo(a)anthracene bis(2-Ethylhexyl) phthalate Butyl benzyl phthalate Chrysene 3,3'-Dichlorobenzidine Pyrene |
| **M-8270-06** | Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Benzo(a)pyrene Dibenz(a,h)anthracene Di-n-octyl phthalate Indeno(1,2,3-cd)pyrene |
| **4-8076** | Carbazole |
| **4-8305-U** | Pyridine |

| Std # | ICV Standard Compounds | | | |
|---|---|---|---|---|
| 506508 | Acenapthene | 2-Chloronaphthalene | 2,4-Dinitrophenol | Naphthalene |
| | Acenaphthylene | 4-Chloro-3-methyl phenol | Dimethyl phthalate | 2-Nitroaniline |
| | Anthracene | 2-Chlorophenol | 2,4-Dinitrotoluene | 3-Nitroaniline |
| | Benzo(a)anthracene | 4-Chlorophenyl phenyl ether | 2,6-Dinitrotoluene | 4-Nitroaniline |
| | Benzo(a)pyrene | Chrysene | Di-n-octyl phthalate | Nitrobenzene |
| | Benzo(b)fluoranthene | Dibenzo(a,h)anthracene | Fluoranthene | 2-Nitrophenol |
| | Benzo(g,h,I)perylene | Dibenzofuran | Fluorene | 4-Nitrophenol |
| | Benzo(k)fluoranthene | Di-n-butyl phthalate | Hexachlorobenzene | N-Nitrosodimethylamine |
| | Butyl benzyl phthalate | 1,2-Dichlorobenzene | Hexachlorobutadiene | N-Nitrosodi-n-propylamine |
| | Bis(2-Chloroethoxy)methane | 1,3-Dichlorobenzene | Hexachlorocyclopentadiene | Pentachlorophenol |
| | Bis(2-Chloroethyl)ether | 1,4-Dichlorobenzene | Hexachloroethane | Phenanthrene |
| | Bis(2-Chloroisopropyl)ether | 2,4-Dichlorophenol | Indeno(1,2,3-cd)pyrene | Phenol |
| | Bis(2-Ethylhexyl)phthalate | 1,2-Diphenylhydrazine | Isophorone | Pyrene |
| | 4-Bromophenyl phenyl ether | Diethyl phthalate | 2-Methylnaphthalene | 1,2,4-Trichlorobenzene |
| | Carbazole | 2,4-Dimethylphenol | 2-Methylphenol | 2,4,5-Trichlorophenol |
| | 4-Chloroaniline | 4,6-Dinitro-2-methylphenol | 4-Methylphenol | 2,4,6-Trichlorophenol |
| S-9076 | Benzoic Acid | N-Nitrosophenylamine | 3,3'-Dichlorobenzidine | Pyridine |
| | Benzyl alcohol | Benzide | Acetophenone | Aniline |
| | 2,6-Dichlorophenol | | | |

| ICV Acceptance Criteria (%R) | | |
|---|---|---|
| Analyte | Low | High |
| 1,2,4-Trichlorobenzene | 35.1 | 59.5 |
| 1,2-Dichlorobenzene | 43.6 | 55 |
| 1,2-Diphenylhydrazine | 36.4 | 66.6 |
| 1,3-Dichlorobenzene | 43.5 | 54.6 |
| 1,4-Dichlorobenzene | 43.6 | 54 |
| 2,4,5-Trichlorophenol | 39.9 | 57.3 |
| 2,4,6-Trichlorophenol | 40.8 | 53.8 |
| 2,4-Dichlorophenol | 33.6 | 60.4 |
| 2,4-Dimethylphenol | 35.5 | 65.8 |
| 2,4-Dinitrophenol | 14.5 | 67.9 |
| 2,4-Dinitrotoluene | 40 | 54.5 |
| 2,6-Dichlorophenol | 33.3 | 59.7 |
| 2,6-Dinitrotoluene | 40.3 | 54.8 |
| 2-Chloronaphthalene | 37.4 | 55.9 |
| 2-Chlorophenol | 43.3 | 56.3 |
| 2-Methylnaphthalene | 40.9 | 56.3 |
| 2-Methylphenol | 35 | 63.4 |
| 2-Nitroaniline | 27.1 | 76.5 |
| 2-Nitrophenol | 37.7 | 56.8 |
| 3,3´-Dichlorobenzidine | 25.4 | 62.6 |
| 3-Nitroaniline | 36.2 | 58.1 |
| 3/4-Methylphenol | 38.3 | 62.7 |
| 4,6-Dinitro-2-methylphenol | 25.3 | 64.8 |
| 4-Bromophenyl phenyl ether | 40.2 | 58.7 |
| 4-Chloro-3-methylphenol | 38.3 | 63 |
| 4-Chloroaniline | 37.8 | 57.6 |
| 4-Chlorophenyl phenyl ether | 31.5 | 67 |
| 4-Nitroaniline | 31.5 | 60.2 |
| 4-Nitrophenol | 10 | 107.2 |
| Acenaphthene | 41.7 | 54.1 |
| Acenaphthylene | 43.4 | 52.3 |
| Acetophenone | 39.7 | 60.1 |
| Aniline | 38.6 | 61.8 |
| Anthracene | 39.8 | 54.5 |
| Benzidine | 10 | 90.7 |
| Benzo[a]anthracene | 41.4 | 54.7 |
| Benzo[a]pyrene | 41.6 | 55 |
| Benzo[b]fluoranthene | 35.3 | 59.5 |
| Benzo[g,h,i]perylene | 27.3 | 64.5 |
| Benzo[k]fluoranthene | 38.6 | 66.5 |
| Benzoic acid | 39.9 | 55.1 |
| Benzyl alcohol | 39.9 | 61.1 |
| Bis(2-chloroethoxy)methane | 40.7 | 56.4 |
| Bis(2-chloroethyl)ether | 41.9 | 58.5 |
| Bis(2-chloroisopropyl)ether | 36.6 | 61.8 |
| Bis(2-ethylhexyl)phthalate | 23.9 | 74.2 |
| Butyl benzyl phthalate | 30.1 | 67.3 |
| Carbazole | 34.9 | 59.8 |
| Chrysene | 40.6 | 57.2 |
| Di-n-butyl phthalate | 41 | 57.1 |
| Di-n-octyl phthalate | 25.2 | 74.7 |
| Dibenz[a,h]anthracene | 30.6 | 61.8 |
| Dibenzofuran | 41 | 53 |
| Diethyl phthalate | 39.1 | 60.6 |
| Dimethyl phthalate | 42.4 | 54.8 |
| Fluoranthene | 37.9 | 57.7 |
| Fluorene | 40.5 | 53.6 |
| Hexachlorobenzene | 34.4 | 61.9 |
| Hexachlorobutadiene | 28.6 | 76.2 |
| Hexachlorocyclopentadiene | 20.3 | 54 |
| Hexachloroethane | 36.6 | 68.2 |
| Indeno[1,2,3cd]pyrene | 30.7 | 61.3 |
| Isophorone | 40.1 | 59.5 |
| N-Nitrosodi-n-propylamine | 31.4 | 73.7 |
| N-Nitrosodimethylamine | 34.2 | 60.4 |
| N-Nitrosodiphenylamine | 39.1 | 55.8 |
| Naphthalene | 43 | 55.1 |
| Nitrobenzene | 36.4 | 65 |
| Pentachlorophenol | 26.4 | 59.6 |
| Phenanthrene | 39.9 | 54.5 |
| Phenol | 39.1 | 60.2 |
| Pyrene | 31.1 | 69 |
| Pyridine | 32.6 | 67.3 |

| LCS/MS/MSD Acceptance Criteria - Waters | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Method 8270 | | | | | Method 625 | | |
| | LCS | | MS/MSD | | | LCS/MS/MSD | | |
| Analyte | Low | High | Low | High | RPD | Low | High | RPD |
| 1,2,4-Trichlorobenzene | 19.7 | 80.9 | 6.56 | 93.1 | 44.8 | 44 | 142 | 44.8 |
| 1,4-Dichlorobenzene | 20.6 | 82 | 12.1 | 93.1 | 39.7 | 20 | 124 | 39.7 |
| 2,4,6-Trichlorophenol | | | | | | 37 | 144 | 30 |
| 2,4-Dichlorophenol | | | | | | 39 | 135 | 30 |
| 2,4-Dinitrotoluene | 23.5 | 95.4 | 18.7 | 96.2 | 36.5 | 39 | 139 | 36.5 |
| 2-Chlorophenol | 31.3 | 81.3 | 5 | 106 | 44.3 | 23 | 134 | 44.3 |
| 2-Nitrophenol | | | | | | 29 | 182 | 30 |
| 4-Chloro-3-methylphenol | 27.2 | 97.7 | 9.03 | 111 | 43.6 | 22 | 147 | 43.6 |
| 4-Nitrophenol | 5 | 56.2 | 5 | 95.2 | 16.6 | 5 | 132 | 16.6 |
| Acenaphthene | 28.6 | 88.6 | 14.5 | 96.6 | 41.3 | 33 | 145 | 41.3 |
| Acenaphthylene | | | | | | 47 | 145 | 30 |
| Anthracene | | | | | | 27 | 133 | 30 |
| 8is(2-chloroethoxy)methane | | | | | | 33 | 184 | 30 |
| Chrysene | | | | | | 17 | 168 | 30 |
| Di-n-butyl phthalate | | | | | | 5 | 118 | 30 |
| Hexachlorobenzene | | | | | | 5 | 152 | 30 |
| N-Nitrosodi-n-propylamine | 15.9 | 119 | 6.65 | 116 | 42.7 | 5 | 230 | 42.7 |
| Naphthalene | | | | | | 21 | 133 | 30 |
| Pentachlorophenol | 5 | 114 | 5 | 135 | 29.2 | 14 | 176 | 29.2 |
| Phenol | 5 | 46.7 | 5 | 7D.4 | 55 | 5 | 112 | 55 |
| Pyrene | 25.3 | 103 | 14.3 | 107 | 37.7 | 52 | 115 | 37.7 |

| LCS/MS/MSD Acceptance Criteria - Solids | | | | | |
|---|---|---|---|---|---|
| | Method 8270 | | | | |
| | LCS | | MS/MSD | | |
| Analyte | Low | High | Low | High | RPD |
| 1,2,4-Trichlorobenzene | 26.4 | 74.6 | 5 | 86.2 | 34.6 |
| 1,4-Dichlorobenzene | 27.8 | 73.3 | 5 | 93.1 | 31.1 |
| 2,4-Dinitrotoluene | 28.6 | 80.D | 12.5 | 83.4 | 29.7 |
| 2-Chlorophenol | 29.4 | 75.7 | 19.6 | 76.5 | 32.0 |
| 4-Chloro-3-methylphenol | 31.7 | 83.0 | 21.1 | 90.3 | 22.4 |
| 4-Nitrophenol | 5 | 121 | 5 | 129 | 17.4 |
| Acenaphthene | 31.0 | 78.8 | 17.1 | 89.1 | 24.5 |
| N-Nitrosodi-n-propylamine | 30.7 | 90.D | 9.25 | 109 | 34.0 |
| Pentachlorophenol | 5 | 106 | 5 | 91.3 | 12.2 |
| Phenol | 28.1 | 77.0 | 10.8 | 87.8 | 31.4 |
| Pyrene | 26.2 | 87.8 | 5 | 116 | 21.4 |

| Initial Demonstration of Capability<br>Spiked at 100 ug/l each | | | | |
|---|---|---|---|---|
| Analyte | Std. Dev. | Std. Dev. Limits | %R | %R Limits |
| 1,2,4-Trichlorobenzene | 7.18 | 28.1 | 59.2 | 44-142 |
| 1,2-Dichlorobenzene | 3.80 | 30.9 | 58.0 | 32-129 |
| 1,3-Dichlorobenzene | 3.57 | 41.7 | 50.9 | 0-172 |
| 1,4-Dichlorobenzene | 4.48 | 32.1 | 54.5 | 20-124 |
| 2,4,5-Trichlorophenol | 2.60 | | 72.1 | 70-130 |
| 2,4,6-Trichlorophenol | 2.99 | | 71.7 | 70-130 |
| 2,4-Dichlorophenol | 5.47 | 26.4 | 72.1 | 39-135 |
| 2,4-Dimethylphenol | 5.58 | 26.1 | 67.7 | 32-119 |
| 2,4-Dinitrophenol | 5.43 | 49.8 | 42.0 | 0-191 |
| 2,4-Dinitrotoluene | 6.09 | 21.8 | 82.4 | 39-139 |
| 2,6-Dichlorophenol | 5.47 | | 72.1 | 70-130 |
| 2,6-Dinitrotoluene | 2.86 | 29.6 | 73.3 | 50-158 |
| 2-Chloronaphthalene | 1.84 | 13 | 56.7 | 60-118 |
| 2-Methylnaphthalene | 2.84 | | 63.4 | 70-130 |
| 2-Methylphenol | 1.79 | | 61.5 | 70-130 |
| 2-Nitroaniline | 3.42 | | 75.6 | 70-130 |
| 2-Nitrophenol | 5.66 | 35.2 | 69.8 | 29-182 |
| 3-Nitroaniline | 4.32 | | 72.3 | 70-130 |
| 3/4-Methylphenol | 2.39 | | 62.0 | 70-130 |
| 4,6-Dinitro-2-methylphenol | 2.71 | 93.2 | 57.8 | 0-181 |
| 4-Bromophenyl phenyl | 3.11 | 23 | 74.9 | 53-127 |
| 4-Chloro-3-methylphenol | 4.20 | 37.2 | 75.6 | 22-147 |
| 4-Chloroaniline | 2.08 | | 88.4 | 70-130 |
| 4-Chlorophenyl phenyl | 6.66 | 33.4 | 78.7 | 25-158 |
| 4-Nitroaniline | 7.91 | | 65.9 | 70-130 |
| 4-Nitrophenol | 2.42 | 47.2 | 23.8 | 0-132 |
| Acenaphthene | 3.95 | 27.6 | 71.0 | 47-145 |
| Acenaphthylene | 3.48 | 40.2 | 70.9 | 33-145 |
| Aniline | 2.50 | | 89.9 | 70-130 |
| Anthracene | 4.75 | 32 | 72.7 | 27-133 |
| Benzo[a]anthracene | 4.14 | 27.6 | 69.0 | 33-143 |
| Benzo[a]pyrene | 4.16 | 39 | 75.6 | 17-163 |
| Benzo[b]fluoranthene | 5.44 | 38.8 | 74.8 | 24-159 |
| Benzo[g,h,i]perylene | 5.98 | 58.9 | 72.5 | 0-219 |
| Benzo[k]fluoranthene | 6.54 | 32.3 | 76.1 | 11-162 |
| Benzoic acid | 6.15 | | 66.2 | 70-130 |
| Benzyl alcohol | 1.79 | | 61.5 | 70-130 |
| Bis(2-chloroethoxy)methane | 4.27 | 34.5 | 74.0 | 33-184 |
| Bis(2-chloroethyl)ether | 0.845 | 55 | 70.3 | 12-158 |
| Bis(2-chloroisopropyl)ether | 2.58 | 46.3 | 72.5 | 36-166 |
| Bis(2-ethylhexyl)phthalate | 3.36 | 41.1 | 63.8 | 8-158 |
| Butyl benzyl phthalate | 3.43 | 23.4 | 63.3 | 0-152 |
| Carbazole | 6.06 | | 75.3 | 70-130 |
| Chrysene | 3.57 | 48.3 | 66.2 | 17-168 |
| Di-n-butyl phthalate | 4.43 | 16.7 | 76.7 | 1-118 |
| Di-n-octyl phthalate | 6.10 | 31.4 | 73.8 | 4-146 |
| Dibenz[a,h]anthracene | 7.48 | 70 | 78.0 | 0-227 |
| Dibenzofuran | 3.91 | | 74.1 | 70-130 |
| Dimethyl phthalate | 3.10 | 23.2 | 74.0 | 0-112 |
| Fluoranthene | 5.11 | 32.8 | 72.4 | 26-137 |
| Fluorene | 5.39 | 20.7 | 75.3 | 59-121 |
| Hexachlorobenzene | 3.67 | 24.9 | 76.1 | 0-152 |
| Hexachlorobutadiene | 3.76 | 26.3 | 54.7 | 24-116 |
| Hexachlorocyclopentadiene | 6.38 | | 70.4 | 70-130 |
| Hexachloroethane | 4.57 | 24.5 | 49.6 | 40-113 |
| Indeno[1,2,3cd]pyrene | 8.54 | 44.6 | 67.4 | 0-171 |
| Isophorone | 6.62 | 63.3 | 92.9 | 21-196 |
| N-Nitrosodi-n-propylamine | 1.79 | 55.4 | 81.7 | 0-230 |
| N-Nitrosodimethylamine | 3.70 | | 49.1 | 70-130 |
| Naphthalene | 4.55 | 30.1 | 64.4 | 21-133 |
| Nitrobenzene | 3.52 | 39.3 | 72.3 | 35-160 |
| Pentachlorophenol | 3.82 | | 79.8 | 70-130 |
| Phenanthrene | 3.93 | 20.6 | 72.7 | 54-120 |
| Phenol | 1.54 | | 34.6 | 70-130 |
| Pyrene | 3.59 | 25.2 | 64.2 | 52-115 |
| Pyridine | 5.96 | | 63.1 | 70-130 |

# STATE OF ILLINOIS

## ENVIRONMENTAL PROTECTION AGENCY

# ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

***SIMALABS INTERNATIONAL - MERRILLVILLE***

***250 WEST 84TH DRIVE***

***MERRILLVILLE, IN 46410***

ACCREDITATION NUMBER #100435

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Janet Cruse

Janet Cruse
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 000620
Expiration Date: 01/30/2003
Issued On: 06/28/2002

# *State of Illinois*
# *Environmental Protection Agency*

Certificate No.: 000620

## Awards the Certificate of Approval

SIMALABS International - Merrillville
250 West 84th Drive
Merrillville, IN 46410

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The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

***Hazardous and Solid Waste, Inorganic***

*1010*

Ignitability

*1311*

TCLP (Organic and Inorganic)

*1312*

Synthetic Precipitation Leaching Procedure

*6010B*

| | | |
|---|---|---|
| Aluminum | Antimony | Arsenic |
| Barium | Beryllium | Cadmium |
| Calcium | Chromium | Cobalt |
| Copper | Iron | Lead |
| Magnesium | Manganese | Molybdenum |
| Nickel | Potassium | Selenium |
| Silver | Sodium | Strontium |
| Thallium | Vanadium | Zinc |

*7060A*

Arsenic

*7131A*

Cadmium

*7421*

Lead

*7470A*

Mercury

*7471A*

Mercury

*7741A*

Selenium

*7841*

Thallium

*9012A*

Cyanide

*9030B*

Sulfides

*9034*

Sulfides

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***Hazardous and Solid Waste, Inorganic*** *9041A*

Hydrogen Ion (pH)

*9045C*

Hydrogen Ion (pH)

*9066*

Phenolics

***Hazardous and Solid Waste, Organic***

*8081A*

| | | |
|---|---|---|
| 1,2-Dibromo-3-chloropropane (DBCP) | 4,4'-DDD | 4,4'-DDE |
| 4,4'-DDT | alpha-BHC | alpha-Chlordane |
| beta-BHC | Chlordane - not otherwise specified | delta-BHC |
| Dieldrin | Endosulfan I | Endosulfan II |
| Endosulfan sulfate | Endrin | Endrin aldehyde |
| Endrin ketone | gamma-BHC (Lindane) | Heptachlor |
| Heptachlor epoxide | Methoxychlor | Toxaphene |

*8082*

| | | |
|---|---|---|
| PCB-1016 | PCB-1221 | PCB-1232 |
| PCB-1242 | PCB-1248 | PCB-1254 |
| PCB-1260 | | |

*8260B*

| | | |
|---|---|---|
| 1,1,1,2-Tetrachloroethane | 1,1,1-Trichloroethane | 1,1,2,2-Tetrachloroethane |
| 1,1,2-Trichloroethane | 1,1-Dichloroethane | 1,1-Dichloroethene |
| 1,1-Dichloropropene | 1,2,3-Trichlorobenzene | 1,2,4-Trichlorobenzene |
| 1,2,4-Trimethylbenzene | 1,2-Dibromo-3-chloropropane (DBCP) | 1,2-Dibromoethane (EDB) |
| 1,2-Dichlorobenzene | 1,2-Dichloroethane | 1,2-Dichloropropane |
| 1,3,5-Trimethylbenzene | 1,3-Dichlorobenzene | 1,3-Dichloropropane |
| 1,4-Dichlorobenzene | 2-Butanone (Methyl ethyl ketone, MEK) | 2-Chloroethyl vinyl ether |
| 2-Hexanone | 2-Nitropropane | 2-Pentanone |
| 4-Methyl-2-pentanone (Methyl isobutyl ketone, I | Acetone | Acetonitrile |
| Acrolein (Propenal) | Acrylonitrile | Benzene |
| Bromobenzene | Bromodichloromethane | Bromoform |
| Bromomethane | Carbon disulfide | Carbon tetrachloride |
| Chlorobenzene | Chlorodibromomethane (Dibromochloromethan | Chloroethane |
| Chloroform | Chloromethane | cis-1,2-Dichloroethene |
| cis-1,3-Dichloropropene | Dichloromethane (Methylene chloride) | Ethyl acetate |
| Ethylbenzene | Isopropylbenzene | Methyl-t-butyl ether |
| m-Xylene | Naphthalene | n-Butanol |
| n-Butylbenzene | o-Xylene | p-Xylene |
| sec-Butylbenzene | Styrene | tert-Butylbenzene |
| Tetrachloroethene | Toluene | trans-1,2-Dichloroethene |
| trans-1,3-Dichloropropene | Trichloroethene | Trichlorofluoromethane |
| Vinyl acetate | Vinyl chloride | |

*8270C*

| | | |
|---|---|---|
| 1,2,4-Trichlorobenzene | 1,2-Dichlorobenzene | 1,2-Diphenylhydrazine |
| 1,3-Dichlorobenzene | 1,4-Dichlorobenzene | 2,4,5-Trichlorophenol |

# *State of Illinois*
# *Environmental Protection Agency*

Certificate No.: 000620

**Awards the Certificate of Approval**

SIMALABS International - Merrillville
250 West 84th Drive
Merrillville, IN 46410

| | | |
|---|---|---|
| ***Hazardous and Solid Waste, Organic*** | *8270C* | 2,4,6-Trichlorophenol |
| 2,4-Dichlorophenol | 2,4-Dimethylphenol | 2,4-Dinitrophenol |
| 2,4-Dinitrotoluene (2,4-DNT) | 2,6-Dichlorophenol | 2,6-Dinitrotoluene (2,6-DNT) |
| 2-Chloronaphthalene | 2-Chlorophenol | 2-Methylnaphthalene |
| 2-Methylphenol | 2-Nitroaniline | 2-Nitrophenol |
| 3,3'-Dichlorobenzidine | 3-Nitroaniline | 4,6-Dinitro-2-methylphenol |
| 4-Bromophenyl phenyl ether | 4-Chloro-3-methylphenol | 4-Chloroaniline |
| 4-Chlorophenyl phenyl ether | 4-Nitroaniline | 4-Nitrophenol |
| Acenaphthene | Acenaphthylene | Acetophenone |
| Aniline | Anthracene | Benzidine |
| Benzo(a)anthracene | Benzo(a)pyrene | Benzo(b)fluoranthene |
| Benzo(g,h,i)perlyene | Benzo(k)fluoranthene | Benzoic acid |
| Benzyl alcohol | Bis(2-chloroethoxy) methane | Bis(2-chloroethyl) ether |
| Bis(2-chloroisopropyl) ether | Bis(2-ethylhexyl) phthalate | Butyl benzyl phthalate |
| Chrysene | Dibenzo(a,h)anthracene | Dibenzofuran |
| Diethyl phthalate | Dimethyl phthalate | Di-n-butyl phthalate |
| Di-n-octyl phthalate | Fluoranthene | Fluorene |
| Hexachlorobenzene | Hexachlorobutadiene | Hexachlorocyclopentadiene |
| Hexachloroethane | Indeno(1,2,3-cd) pyrene | Isophorone |
| m-Cresol (3-Methylphenol) | Naphthalene | Nitrobenzene |
| N-Nitrosodimethylamine | N-Nitrosodi-n-propylamine | N-Nitrosodiphenylamine |
| p-Cresol (4-Methylphenol) | Pentachlorophenol | Phenanthrene |
| Phenol | Pyrene | Pyridine |
| *8310* | | |
| Acenaphthene | Acenaphthylene | Anthracene |
| Benzo(a)anthracene | Benzo(a)pyrene | Benzo(b)fluoranthene |
| Benzo(g,h,i)perylene | Benzo(k)fluoranthene | Chrysene |
| Dibenzo(a,h)anthracene | Fluoranthene | Fluorene |
| Indeno(1,2,3-cd) pyrene | Naphthalene | Phenanthrene |
| Pyrene | | |
| ***Wastewater, Inorganic*** | | |
| *SM2510B,18Ed* | | |
| Specific Conductance | | |
| *SM3500Cr-D,18Ed* | | |
| Chromium VI | | |
| *SM4500CL-B,18Ed* | | |
| Chloride | | |
| *SM4500CN-CE18Ed* | | |
| Cyanide | | |
| *SM4500CN-CG18Ed* | | |
| Cyanide-amenable to chlorination | | |
| *SM4500O-C,18Ed* | | |
| Oxygen - Dissolved | | |
| *SM5210B,18Ed* | | |
| Biochemical Oxygen Demand (BOD) | Carboneous Biochemical Oxygen Demand (CB | |

# *State of Illinois*
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SIMALABS International - Merrillville
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---

***Wastewater, Inorganic*** *USEPA110.2*

Color

*USEPA130.2*

Hardness

*USEPA150.1*

Hydrogen Ion (pH)

*USEPA160.1*

Residue (TDS)

*USEPA160.2*

Residue (TSS)

*USEPA160.3*

Residue (Total)

*USEPA160.4*

Residue (Volatile)

*USEPA1664RA*

Oil and Grease

*USEPA170.1*

Temperature

*USEPA200.7*

| | | |
|---|---|---|
| Aluminum | Antimony | Arsenic |
| Barium | Beryllium | Boron |
| Cadmium | Calcium | Chromium |
| Cobalt | Copper | Iron |
| Lead | Magnesium | Manganese |
| Molybdenum | Nickel | Potassium |
| Selenium | Silica | Silver |
| Sodium | Thallium | Tin |
| Vanadium | Zinc | |

*USEPA206.2*

Arsenic

*USEPA213.2*

Cadmium

*USEPA239.2*

Lead

*USEPA245.1*

Mercury

*USEPA270.2*

Selenium

*USEPA279.2*

Thallium

*USEPA310.1*

Alkalinity

*USEPA330.5*

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| | | |
|---|---|---|
| ***Wastewater, Inorganic*** | *USEPA330.5* | Chlorine |
| *USEPA335.2* | | |
| Cyanide | | |
| *USEPA340.2* | | |
| Fluoride | | |
| *USEPA350.1* | | |
| Ammonia | | |
| *USEPA350.2* | | |
| Ammonia | | |
| *USEPA351.3* | | |
| Total Kjeldahl Nitrogen | | |
| *USEPA353.2* | | |
| Nitrate-Nitrite (sum) | | |
| *USEPA354.1* | | |
| Nitrite | | |
| *USEPA365.1* | | |
| Orthophosphate (as P) | | |
| *USEPA365.3* | | |
| Phosphorus | | |
| *USEPA375.4* | | |
| Sulfate | | |
| *USEPA405.1* | | |
| Biochemical Oxygen Demand (BOD) | | |
| *USEPA410.4* | | |
| Chemical Oxygen Demand (COD) | | |
| *USEPA420.2* | | |
| Phenolics | | |
| ***Wastewater, Organic*** | | |
| *USEPA608* | | |
| 4,4'-DDD | 4,4'-DDE | 4,4'-DDT |
| Aldrin | alpha-BHC | beta-BHC |
| Chlordane | delta-BHC | Dieldrin |
| Endosulfan I | Endosulfan II | Endosulfan sulfate |
| Endrin | Endrin aldehyde | gamma-BHC (Lindane) |
| Heptachlor | Heptachlor epoxide | PCB-1016 |
| PCB-1221 | PCB-1232 | PCB-1242 |
| PCB-1248 | PCB-1254 | PCB-1260 |
| Toxaphene | | |
| *USEPA610* | | |
| Acenaphthene | Acenaphthylene | Anthracene |
| Benzo(a)anthracene | Benzo(a)pyrene | Benzo(b)fluoranthene |
| Benzo(g,h,i)perylene | Benzo(k)fluoranthene | Chrysene |
| Dibenzo(a,h)anthracene | Fluoranthene | Fluorene |

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SIMALABS International - Merrillville
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Merrillville, IN 46410

***Wastewater, Organic***

Naphthalene

*USEPA624*

1,1,1-Trichloroethane
1,1-Dichloroethane
1,2-Dichloroethane
1,4-Dichlorobenzene
Benzene
Bromomethane
Chloroethane
cis-1,3-Dichloropropene
Ethylbenzene
trans-1,2-Dichloroethene
Trichlorofluoromethane

*USEPA625*

1,2,4-Trichlorobenzene
1,4-Dichlorobenzene
2,4-Dimethylphenol
2-Chloronaphthalene
3,3'-Dichlorobenzidine
4-Chlorophenyl phenyl ether
Acenaphthylene
Benzo(a)anthracene
Benzo(g,h,i)perylene
Bis(2-chloroethoxy) methane
Chrysene
Dimethyl phthalate
Fluoranthene
Hexachlorobutadiene
Indeno(1,2,3-cd) pyrene
Nitrobenzene
N-Nitrosodiphenylamine
Phenol

*USEPA610*

Phenanthrene

1,1,2,2-Tetrachloroethane
1,1-Dichloroethene
1,2-Dichloropropane
2-Chloroethylvinyl ether
Bromodichloromethane
Carbon tetrachloride
Chloroform
Dibromochloromethane
Tetrachloroethene
trans-1,3-Dichloropropene
Vinyl chloride

1,2-Dichlorobenzene
2,4,6-Trichlorophenol
2,4-Dinitrotoluene (2,4-DNT)
2-Chlorophenol
4-Bromophenyl phenyl ether
4-Nitrophenol
Anthracene
Benzo(a)pyrene
Benzo(k)fluoranthene
Bis(2-chloroethyl) ether
Dibenzo(a,h)anthracene
Di-n-butyl phthalate
Fluorene
Hexachlorocyclopentadiene
Isophorone
N-Nitrosodimethylamine
Pentachlorophenol
Pyrene

Indeno(1,2,3-cd) pyrene

Pyrene

1,1,2-Trichloroethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
Acrylonitrile
Bromoform
Chlorobenzene
Chloromethane
Dichloromethane (Methylene chloride)
Toluene
Trichloroethene

1,3-Dichlorobenzene
2,4-Dichlorophenol
2,6-Dinitrotoluene (2,6-DNT)
2-Nitrophenol
4-Chloro-3-methylphenol
Acenaphthene
Benzidine
Benzo(b)fluoranthene
Benzyl butyl phthalate
Bis(2-ethylhexyl) phthalate
Diethyl phthalate
Di-n-octyl phthalate
Hexachlorobenzene
Hexachloroethane
Naphthalene
N-Nitrosodi-n-propylamine
Phenanthrene

SIMALABS International
Data Review Checklist – Metals

Run ID: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| PDS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

- The control is biased high bias yet the analyte is "non-detectable" in the sample.
- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

SIMALABS International
Data Review Checklist – Wet Chemistry

Run ID: _____ Analyte: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD accuracy criteria met? | ☐ Yes ☐ No ☐ NA | |
| MSD / DUP precision criteria met? | ☐ Yes ☐ No ☐ NA | |
| PDS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

- The control is biased high bias yet the analyte is "non-detectable" in the sample.
- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

SIMALABS International
Data Review Checklist – GC

Run ID: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Endrin/DDT breakdown criteria met? (Pesticide analyses only) | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| SURR acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |
| Acceptable confirmation performed on 2nd column? | ☐ Yes ☐ No ☐ NA | |
| Manual integrations appropriately performed and identified? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

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- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

SIMALABS International
Data Review Checklist – GC/MS

Run ID: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Tune criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ISTD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| SURR acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |
| Manual integrations appropriately performed and identified? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

Data evaluated as "No" can be reported without a Case Narrative if any of the following apply (but a CAR is still required).

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- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

SIMALABS International
Data Review Checklist – HPLC

Run ID: _____ Analyst: _____

**1st Level Technical Review**

| Review Element | Evaluation * | Comments (use this space as needed) |
|---|---|---|
| Calibration curve acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| ICV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| CCV acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MB acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| LCS acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| MS/MSD acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| SURR acceptance criteria met? | ☐ Yes ☐ No ☐ NA | |
| Analyses checked for carryover contamination? | ☐ Yes ☐ No ☐ NA | |
| Manual integrations appropriately performed and identified? | ☐ Yes ☐ No ☐ NA | |

* Evaluations are applicable to target analytes only. Any "No" answer requires a Comment and the initiation of a Corrective Action Report (CAR) if the data is to be used.

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- Blank contamination yet the analyte measured in the sample is "non-detectable" or ≥ 10X the blank contamination.
- Unacceptable MS/MSD recovery but the sample concentration is ≥ 5X the concentration of the spike added.

Data evaluated as "No" may be reported if any of the following apply, however a CAR and Case Narrative are required. Case Narratives are generated by the QA department prior to data validation in the LIMS.

- Unacceptable MS/MSD recoveries handled in accordance with the MS/MSD Corrective Action Flowchart included in the QAP.
- Insufficient sample, holding time, or turnaround time available for reanalysis.
- Data meets the needs of the client (as per Project Manager).

*I certify that the above assessment has been performed and the analyses were performed according to the operating conditions and procedures contained in the current version of the Standard Operating Procedure.*

Initials: _____ Date: _____

**2nd Level Technical Review**

Above assessment accurate ☐ Yes ☐ No
Data accurate in LIMS ☐ Yes ☐ No
If "No', list unacceptable evaluation(s): _____

LIMS QA Validation performed ☐ Entire Run ☐ Partial Run ☐ No samples validated
Initials: _____ Date: _____

**Baker Environmental, Inc.**
*A Unit of Michael Baker Corporation*

# Richard M. Spitaler P.G.

Environmental Geologist

**Years with Baker:** 4
**Years with Other Firms:** 7

**Education**
Northeastern Illinois University
Masters Degree Program
Geography and Environmental Sciences

Southern Illinois University
B.A., Geology, 1981

University of Illinois
B.A., Psychology, 1978

**Registrations**
Professional Geologist
Indiana, Illinois

National Groundwater Association Member

## General Qualifications

Mr. Spitaler is a Professional Geologist with experience in the Environmental and Engineering Consulting Fields. He has completed a variety of environmental projects in areas ranging from investigation to decommissioning oversight. Project focus has included: groundwater quality issues (from investigation to remediation); independent technical reviews; regulatory permitting and compliance; environmental issues surrounding real estate transactions; and, underground storage tank closures.

## Experience

- **RCRA Facility Investigations**. Implementation of a comprehensive groundwater quality investigation (RCRA agreed order) for two major steel mills in Northwest Indiana. Duties included oversight of the installation of monitoring well networks at several hazardous waste facilities, characterization of soils, collection of soil and groundwater samples, coordination of site activities and assisted in data analysis and presentation for regulatory reporting.

- **IDEM Contract Services.** Provided Remedial Action implementation at an IDEM Superfund Section investigation/monitoring program in north central Indiana, assisted with O&M at a long-term IDEM LUST remediation project, and provided technical review of documents submitted the IDEM Voluntary Remediation Program (VRP) at numerous sites across Indiana. Also served as a Project Geologist for a site under the IDEM VRP located in Lafayette, Indiana that was completed to receipt of a Covenant-not-to-Sue.

- **Hydrogeological and Subsurface Investigations.** Supervised and managed numerous geological investigations at commercial, industrial, and government sites. This has included the supervision of monitoring programs, SVE pilot testing, in-situ biodegradation pilot testing, and rising/falling head tests. Performed numerous subsurface drilling investigations for sites with contaminated soil and/or groundwater. Duties included split spoon sampling, sand sampler sampling, shelby tube sampling, lithologic descriptions, cone penetration testing, Geoprobe, geochemical, waste, sediment, surface water, groundwater, soil, rock and analytical sample collection, health and safety supervisor, monitoring, geotechnical investigations, monitoring, multiple casing and recovery well installation, strict decontamination and documentation procedures, air and water surging well development, hydro-washing well development, well sampling, quality control sampling, aquifer testing, natural attenuation screening, etc.

- **Underground Storage Tanks.** Managed site investigations and the decommissioning of underground storage tanks (USTs). Familiar with all

phases of UST work, which included 2 years of UST related work experience at a major steel mill. Drafted the required reports and worked with state and local agencies to achieve regulatory compliance in the most cost effective manner.

- **Environmental Assessments.** Worked on Phase I and Phase II environmental assessments at various industrial and commercial facilities. Responsibilities have included: record research and record review, interpretation of aerial photographs as well as topographic and geologic maps, and report writing.

- **Environmental and Regulatory Compliance Sampling.** Supervised numerous sampling investigations, authored sampling plans, conducted soil, wipe, lagoon, tank, drum, smokestack and waste water treatment sampling, written QA/QC protocols, and developed health and safety plans.

- **Asbestos Remediation.** Have two years experience with asbestos abatement projects. Responsibilities have included: microscopic analysis of ACM, air monitoring for regulatory compliance, site inspections, and monitoring the health and safety of workers.

- **Waste Water Treatment.** Have worked on several waste water treatment projects. Responsibilities have included sludge densification pilot testing, settling rate and flow rate calculations, environmental compliance sampling, and NPDES permitting.

- **Environmental Project Audits.** Provided independent technical review of the remedial investigation, remedial approach, and remedial system design at UST sites for the State of Wisconsin. These audits assessed the technical merit and cost effectiveness of the UST related work performed by the primary responsible parties environmental consultants. In addition, provided independent technical review and evaluation of consultants remedial practices for the Department of Commerce's legal department.

- **Regulatory Compliance.** Worked on the preparation of reports and applications for NPDES permitting, SARA TITLE III, sections 311, 312, and 313 reporting. Clean Air permitting, and Consent Decree compliance.

- **Department of Transportation**. Two environmental contracts with the Illinois Department of Transportation that included the generation of work plans, participation in weekly progress meetings, environmental screening and sampling, coordination of storage, trucking, and disposal of soil and water waste, and the supervision of environmental operations during construction within areas of suspected contamination.

# James D. Peyton

## Senior Environmental Geologist

**Years with Baker:** 11
**Years with Other Firms:** 0

**Education**
Indiana University
Bloomington, Indiana
B.A., Geology, 1990

**Registration**
AHERA Building Inspector
Troxler Nuclear Testing Equipment Operator
Wisconsin UST Site Assessor

Professional Geologist licensed in Indiana and Illinois

**Highlights**

VRP Knowledge.

Reduced costs through use of advancements in direct push and field analytical services.

RISC Training.

IDEM received highly favorable reviews by both interagency and USEPA reviewers.

Mr. Peyton is a senior geologist in the site characterization section of the Merrillville, Indiana Regional Office at Baker Environmental, Inc. He has completed a variety of environmental projects in areas ranging from investigation to remediation and construction oversight. Project focus has included groundwater quality issues (from investigation to remediation), solid and hazardous waste management, environmental issues surrounding real estate transactions, construction management, and underground storage tank closures.

During the course of an eleven year relationship with the Indiana Department of Environmental Management (IDEM), he has provided technical assistance on numerous projects and committees including: community relations, project reviews, specific technical/methodology questions for assessment, investigations and remediation, research and vender searches, policy impact discussions, alternative policy cost comparisons, inter and intra-agency relations, interpretation of agency regulation/guidance, project funding, appearance at public meetings, and enforcement case litigation support.

**Experience**

- Project Manager/Geologist for the IDEM Voluntary Remediation Program (VRP) at numerous sites across Indiana for technical review of documents submitted to IDEM, technical support, field inspections, confirmation sampling and participation as technical consultant at project meetings.

- Project Field Manager/Geologist for an IDEM Superfund Section Remedial Design and Remedial Action site in north central Indiana. Developed work plans; conducted soil and groundwater sampling; via GeoProbe, monitoring well sampling, and well installation; mobile, State contract, and private laboratory coordination; groundwater modeling; natural attenuation monitoring; reporting, and community relations.

- Project Geologist for RISC guidance development, committees, and reviews: worked with IDEM for develop a cost comparison of proposed RISC rules to other State programs including statistical sampling method/judgmental sampling comparisons; received the same four-day RISC training as IDEM staff; and, Team Leader for technical reviews of RISC documents received from IDEM for comment.

- Project Manager for a major site characterization at a manufacturing facility in South Bend, Indiana for IDEM. Included preparation of project plans (QAPP, SAP & HASP), the implementation of these plans in the field (off-site well survey and sampling, soil gas survey, cone penetrometer testing (CPT) soundings and groundwater sampling, auger borings, HydroPunch sampling, well installation, soil and groundwater sampling), the coordination of analytical data validation, the interpretation of investigation

results, conducted project progress meetings with IDEM technical support and management staff, report preparation and development of remedial alternative options. Performed additional services for IDEM including pumping test, groundwater modeling, gross cost remedial alternative evaluation, and review of municipal water system. Provided IDEM and municipal water department with technical assistance during continuing evaluation of remedial/water supply alternatives, technical review of facility owner and USEPA documents, and National Priority List Scoring evaluation review. Ongoing activities include: free product delineation, migration monitoring, mitigation alternative evaluations and implementation, and providing technical support to IDEM.

Reduced IDEM O&M costs with system upgrades.

- Project Geologist for the technical operation, maintenance, and monitoring of an air stripper, above ground vapor filtration and carbon filtration system in northern Indiana for IDEM. Researched, designed and implemented successive system upgrades that increased overall operating efficiency and lowered operational expenditures. Supervised and coordinated all construction activities including grading site to design specification; installation of geo-membrane liner; distribution piping; distribution pea gravel and filter material; installation of the air stripper and system plumbing; and system maintenance. Coordinated the removal, disposal and replacement of clogged packing material from multiple air strippers. In addition, an Operation and Maintenance Manual, construction, and system operations reports were prepared.

IDEM received praise for effective application.

- Project Geologist for the installation of ventilation systems in residences impacted by petroleum vapors in northern Indiana for IDEM. Coordinated the installation with homeowners, system design, contractors, state agency representatives, supervised the system installations and modifications.

Long-term working relationship with IDEM.

- Project Manager/Geologist for leaking underground storage tank investigation sites in Indiana for IDEM. Projects consist of work plans, soil gas surveys, installation of monitoring wells, hydrogeological studies, remediation system design, operations and maintenance, and local community involvement.

RCRA Agreed Order for hundreds of SWMUs and AOCs.

- Project Manager/Geologist for ongoing projects under a RCRA Agreed Order addressing hundreds of SWMUs and AOCs at a northern Indiana facility. Responsible for work plan development, response to USEPA comments, implementation of work plans, conceptual site models, visual inspections, historical research, technical support during agency negotiations, coordination with multiple consultants, sub-contractors, and facility managers, data summaries, evaluations and reports.

No Further Action for 57 RCRA SWMU/AOCs.

- Project Geologist for a completed RCRA Facility Investigation Phase 1 for 64 SWMU/AOCs at a northern Indiana facility. Developed/revised work plans, response to USEPA comments, implementation of work plans, technical support during agency negotiations, coordination of multiple sub-contractors, facility managers, data summaries, evaluations and 23 volume reports. Project Manager for remaining SWMU/AOC Phase II activities.

Reduced sampling costs through negotiations with VRP.

- Project Manager/Geologist for sites under the IDEM VRP located in central Indiana. Included VRP applications, development of work plans, multi-matrix field investigation, aquifer testing, reports, determination of program approach, groundwater modeling, risk assessment, natural attenuation evaluations, response to comments, and client/VRP negotiations/meetings.

UST/LUST closures.

- Project Geologist/Field Manager for over thirty underground storage tank closures in Indiana and Illinois. Included site supervision of removal activities, regulatory reporting, permitting and notification, collection of confirmatory samples, review of analytical results, research and coordination of altemative off-site disposal options, tracking and review of client contractor financial disputes, and closure reports. Supervised and tested backfill material under strict construction specifications and prepared and implemented multiple site restoration specifications.

Large scale geotechnical investigations.

- Project Geologist for a large-scale geotechnical drilling and testing program for landfill design at two major steel mills in Northwest Indiana. Included oversight, installation of numerous monitoring wells, detailed soil characterization, vane shear testing, geotechnical in-situ testing, supervision of multiple drill rigs and personnel, and the collection of soil samples for geotechnical parameters and groundwater samples for analysis.

Client avoided remedial costs through innovative analytical data evaluations.

- Project Geologist for the investigation of a large pipeline release adjacent to a state wildlife refuge, located near Griffith, Indiana. Conducted soil, groundwater and free product sampling on an accelerated schedule. Hydrocarbon fingerprinting was compared to product usage records from pipelines operated by three companies.

Large scale RCRA groundwater programs

- Project Geologist for the implementation of a comprehensive groundwater quality investigation (RCRA agreed order) for a major steel mill in Northwest Indiana. Included oversight, installation of monitoring well networks at several hazardous waste facilities, characterization of soils, collection of soil, sediment, surface water, waste and groundwater samples for geotechnical and analytical testing, coordination geophysical surveys, supervision of site activities, QA/QC, data analysis and presentation for regulatory reporting.

Alterations of water supply system eliminated need for costly remediation system.

- Project Field Manager for a sustained pilot test at a central Indiana municipal water supply. Alternative pumping scenarios were conducted to evaluate water treatment operations at the facility. The pilot study demonstrated through controlled groundwater pumping that contamination could be remediated and was protective of public service water quality.

Client was able to transfer operations to new owner due effective treatment system.

- Project Geologist for the technical operation, maintenance, and monitoring of a bio-remediation system in Hammond, Indiana. The system was designed to extract groundwater from a network of horizontal extraction trenches, introduce commercially produced hydrocarbon degrading bacteria and nutrients, and re-injection into dispersion trenches or spray irrigation of impacted soils. Conducted cleanup objective verification sampling of treatment cells and periodic system operations sampling.

MGP fractured bedrock migration and remediation.

- Project Geologist for the delineation and remediation of coal tar (MGP site) migration through a fractured bedrock formation in western Illinois. Conducted remedial alternative evaluation for client operated remedial action.

Client reduced costs by consolidating operations.

- Quality Control Officer for a RCRA lagoon closure at a steel finishing mill located in central Illinois. Included inspection of construction activities to meet strict specifications during sludge stabilization, transportation, and deposition into an associated site landfill, final inspection of lagoon prior to closure, density and moisture verification testing during placement of backfill material, and final inspection of restoration activities. Responsibilities also included trouble shooting during ongoing site construction activities project progress meetings with the client and subcontractor. Designed and conducted a groundwater monitoring plan under the IEPA Landfill permit and reporting to the Agency.

Favorable decision for residents and IDEM.

- Provided witness testimony during a civil suit in Indiana, associated with an ongoing State of Indiana enforcement case at a petroleum release site. Services included litigation support during pre-trial discussions and courtroom testimony. Provided testimony on site investigations, local aquifer characteristics, probable sources of the contested soil and groundwater contamination, plume migration history, State plume interception actions, and site observations. The court case was decided in favor of the client and the State of Indiana in a subsequent Summary Judgement decision.

- Geologist/Supervisor for environmental contracts with the Illinois DOT. Generation of work plans, progress meetings, sampling and disposal of soil/water, and supervision of operations within areas of contamination adjacent to ecologically sensitive areas.

Ecological and human exposure evaluation.

- Project Geologist for the evaluation of environmental impacts from with glass slurry disposal in landfills and coal-mine backfill in Pennsylvania. Groundwater, and coal-mine seeps, surface and surface soil exposure pathways for ecological and human exposure were evaluated; remedial measures were recommended and implemented.

Performance of real time aquifer evaluation prevented costly remobilization.

- Project Geologist at former missile sites for the Army Corps of Engineers natural attenuation parameter and aquifer testing at sites in Nebraska, Wyoming and Colorado.

Drilling Experience.

- Performed numerous subsurface drilling investigations for sites with contaminated soil and/or groundwater. Duties included split spoon sampling, sand sampler sampling, shelby tube sampling, lithologic descriptions, cone penetration testing, Geoprobe, geochemical, waste, sediment, surface water, groundwater, soil, rock and analytical sample collection, health and safety supervisor and monitoring, geotechnical investigations, monitoring, multiple casing and recovery well installation, strict decontamination and documentation procedures, air and water surging well development, hydro-washing well development, well sampling, quality control sampling, aquifer testing, natural attenuation screening, etc.

**Baker Environmental, Inc.**
*A Unit of Michael Baker Corporation*

# Thomas P. Noble

Environmental Scientist

**Years with Baker:** 1
**Years with Other Firms:** 24

**Education**

College of DuPage
General Studies
Illinois Institute of Technology
Mechanical Engineering
Preparation

**Registration**

IDPH-AHERA Building Inspector, IDPH-Lead Based Inspector and Risk Assessor

## General Qualifications

Mr. Noble is an Environmental Scientist in the Midwest Region Office of Baker Environmental, Inc. with extensive experience in environmental consulting and site remediation. He has completed a variety of environmental projects from investigation, to remediation construction oversight, and operations and maintenance (O&M).

## Experience

- Performed site inspections and regulatory compliance reviews for a national client at existing and new sites, and assisted with local agency reviews.

- Performance of Phase I and Phase II Environmental Site Assessments throughout Illinois, Indiana, Michigan, Texas, New Jersey, New York, Florida, Tennessee, Virginia, Ohio, California, Idaho, and Washington. Some projects included Asbestos Building Inspections and Lead Based Paint Inspections and Risk Assessments. Licensed in Illinois, Indiana, Wisconsin, and Washington.

- Preliminary Site Assessments for wireless telecommunication antenna location. Assembled team to perform assessments, coordinated activities with client, provided QA/QC with direction for limited Phase II sampling activities managed performance of sample collection and laboratory subcontract.

- IEPA SRP in-situ lead stabilization and removal project at a shopping mall. Removed approximately 3,000 tons of stabilized soil for disposal. Completed project and obtained NFR letter for the Property owner.

- Managed and performed sampling activities at former PCB capacitor manufacturing facility for segregation of materials during demolition of manufacturing building prior to incineration activities in Thermal Desorption Unit.

## CERCLA Sites

- Assisted with Remedial investigative activities prior to construction. Activities included construction of work platform and slurry wall within "Exclusion Zone." After remedial activities had established "clean line of travel, established RCRA landfill cap.

- Performed and managed activities related to Phase I and Phase II Environmental Site Assessments to ASTM Standard of Practice E1527-97, and Asbestos building inspections and bulk sample collection activities per AHERA and 40 CFR 763 protocols and regulations.

- Performed a baseline assessment activities at Fernald Environmental Management Project. Areas of responsibility included TSCA/PCB management and handling of wastes for storage and disposal, sampling, and analytical procedures.

- Construction Manager for the remediation of PCB contaminated sites on company-owned and privately owned properties. Activities included managing daily waste handling operations and waste minimization efforts at various company service centers and four electric generating stations.

- Supervisor of crews assigned routine and emergency maintenance activities for equipment related to the O&M and operation of steam generating equipment, turbines, generators, and related auxiliary equipment.

- Responsible for O&M and operation of air pollution control equipment including; electrostatic precipitators, fly ash control equipment, and related fugitive dust collection equipment. Performed troubleshooting activities for systems and oversight of maintenance activities.

- Responsible for O&M and operation of wastewater treatment collection, boiler demineralization system and boiler water analytical quality.

**Baker Environmental, Inc.**
*A Unit of Michael Baker Corporation*

# Margaret C. James

Environmental Scientist

**Years with Baker:** 4.5
**Years with Other Firms:** 0

**Education**
Saint Joseph's College
B.S., Environmental Science,
1996

## General Qualifications

Ms. Margaret (Peggy) James is an assistant environmental scientist with experience in ASTM Standard Phase I and II Environmental Site Assessments, ecological surveys, and environmental database management, database design, and SARA Title III Compliance Work.

## Experience

**Database Design**

- Designed and implemented several Microsoft Access databases to assist in tracking and responding to public comments, and record sources for the Indiana Department of Transportation Route 231 Relocation project.

- Designed and implemented a Microsoft Access database to assist in tracking Fugitive Dust sources at a major steel manufacturing facility in Northwest Indiana. Database was used in compiling fugitive dust sources, information regarding vehicular traffic, and road conditions which contribute to fugitive dust releases.

**Database Management**

- Analytical Data manager to a major steel manufacturer during the coarse of RCRA corrective action being performed in Northwest, Indiana. Responsibilities include data tracking of many different sampling activities occurring at various locations at the facility. Create and maintain a sample-tracking database that encompassed sample collection information, relevant field parameters, information and chain of custody information. Create and maintain a unique identifier system to correspond with each sampling location and depth for various sample media including surface soil, subsurface soil, surface water, waste, groundwater, and sediment. Create and maintain uniform field parameter reporting spreadsheets. Facilitate smooth accurate transfer of sample from field locations to laboratory under proper chain of custody and assurance that all necessary analysis is performed for any given sample. Transfer electronic laboratory data and validation of that data from text format for final input into Terra Base.

- Provided data management service for Bayer Corporation, Elkhart; Indiana. Assisted in developing several Microsoft Excel spreadsheets and Microsoft Access Databases encompassing topics of equipment tracking, waste disposal activities, hazardous waste manifests, and tracking of asbestos removal activities.

- Provided data management service and research knowledge to a major steel manufacturer in Northwest, Indiana. Created and maintained Microsoft Excel spreadsheets to assist in the development of a conceptual site model detailing spills and releases throughout the facilities operation. Assisted in the development of a comprehensive listing of areas of environmental interest contained within the facility.

**Baker Environmental, Inc.**
*A Unit of Michael Baker Corporation*

**Phase I / II Environmental Site Assessments**

- Ms. James has conducted numerous Phase I site assessments in Indiana. Work on ESAs involved: conducting site visits, recognizing environmental concerns (past and present), reviewing records and previous reports, researching property title transfers and historical usages, conducting interviews, making recommendations and writing reports.

**Environmental Sampling Experience**

- Provided RCRA RFI services for two major steel manufacturers in the Midwest. Assisted in the groundwater-sampling program including well development, purging, and sampling as well as hydropunch groundwater sampling. . Groundwater sampling program included well development, low flow purge, collection of field parameter including, pH, specific conductivity, dissolved oxygen, oxidation-reduction potential, turbidity, and temperature. Additional services included ecological field survey, and soil and waste stream sampling. Assisted with laboratory data validation management and QA/QC activities.

- Performed hazardous waste characterization and surface water and sediment sampling to a major steel manufacturer in Northwest, Indiana. Collected several different waste stream samples for various analyses.

- Performed surface soil and groundwater sampling for a manufacturing facility in Central Indiana. Sampled multiple riverbank locations using hand augers and stainless steel spoons. Groundwater samples were collected at a low flc purge. Field parameters were collected on sight using Horiba U-22 model and HACH total iron field kit.

- Performed groundwater sampling at a chemical manufacturing facility in Charleston, West Virginia. Sampling consisted of twenty-four flush mount wells using tephlon bailers. Samples were then labeled and documented for Chain of Custody's and shipped offsite to an independent laboratory.

# CHRISTOPHER D. BARTOSZ, P.L.S.

*Director of Surveying*

**PROFESSIONAL EXPERIENCE:**

V3 Companies, Ltd.
Director of Surveying
2001 - present

V3 Companies, Ltd.
Project Manager of Land Surveying Services
1999-May 2001

Cowhey, Gudmundson, Leder
Project Manager of Land Surveying Services
1993-1999

Bollinger, Lach & Associates
Crew Chief and Project Manager of Land Surveying Services 1986-1993

**EDUCATION:**

Iowa State University
Ames, Iowa

Associates Degree in Science
College of DuPage
Glen Ellyn, Illinois

**REGISTRATION:**

Professional Land Surveyor, Illinois

**PROFESSIONAL ASSOCIATIONS:**

Illinois Professional Land Surveyor Association

Mr. Bartosz has over seventeen years experience in all phases of land surveying. His project management work includes large-scale commercial and residential land development surveying projects from site development through final subdivision, construction and post-construction phases, as well as commercial land title and construction surveying. Mr. Bartosz has performed location studies for municipalities, topographic and environmental studies. He has been involved in major land acquisition and route surveying projects for the Illinois Department of Transportation and the Illinois State Tollway Authority.

## PROJECT INVOLVEMENT

**Soldier Field, Chicago, Illinois**- Project Manager responsible for the 160 acre Stadium and Park renovation. The scope of surveying services included highly accurate surveying methods to locate 1400 support columns and other key structural elements underneath the Stadium, as well as key architectural elements on the Stadium roof and colonnades. The project also consisted of an enormous amount of data collection, gathered by Global Positioning Systems and traditional surveying methods of the Stadium parking lot, adjacent Museum parking lots, Burnham Harbor and McCormack Place infrastructure. The resulting product consisted of a 30-sheet topographic and boundary survey showing all of the aforementioned information in great detail.

**Braidwood Nuclear Power Plant, Braidwood, Will County, Illinois**- Project Manager responsible for a thirteen square mile land title survey of a nuclear facility. With the use of Global Positioning Systems and traditional surveying methods, V3 was able to perform thirteen square miles of boundary surveying, as well as locating improvements within and adjacent to the aforementioned facility. Services also included the creation of many permanent and temporary easement exhibits.

**Chicago Premium Outlets, Aurora, Illinois**- Project Manager responsible for a 230-acre commercial/industrial land title survey and topographic map. With the combination of Global Positioning Systems and traditional surveying methods, V3 was able to produce a highly detailed, accurate Governmental section land title survey and topographic map, which included numerous amounts of complicated easements and restrictions.

# CHRISTOPHER D. BARTOSZ, P.L.S.

*Director of Surveying*

**PROJECT INVOLVEMENT**

**CVS Pharmacy Chain, Chicagoland, Illinois**- Project Manager responsible for the surveying services for over twenty sites throughout Chicagoland. The services included highly detailed land title surveys and topographic maps, plat of vacations, plat of easements, plat of consolidations and plat of subdivisions. We are presently managing the construction of several of these sites.

**Illinois Department of Transportation Land Acquisition – District 2 -** Surveyor-in-charge of an eight-mile land acquisition project for a state highway between Tampico, Illinois and Dixon, Illinois.

**Cambridge at Carillon, Will County, Illinois-** Surveyor-in-charge from start to finish of a 700-acre golf course and senior citizen residential development. The scope of surveying services for this project included traditional surveying methods to establish horizontal and vertical control for the 3D locations of all improvements for topographic studies. Additionally, Chris prepared multiple plats for the project and was the project manager for construction and post-construction phases.

**Falling Water Subdivision, Burr Ridge, Illinois** – Surveyor-in-charge from start to finish of this 120-acre upscale residential development. The scope of surveying services for this project included traditional surveying methods to establish horizontal and vertical control for the 3D location of all improvements and for boundary and topographic studies. In addition, Chris prepared multiple plats for the project and was the project manager for construction and post-construction phases.

# R. DAMON LEE, CHMM, REA

*Director of Environmental Redevelopment*

**PROFESSIONAL EXPERIENCE:**
V3 Consultants, Director of Environmental Redevelopment 1999-Present

Montgomery Watson, Principal Environmental Investigation & Remediation
1985-1999

Ohio Environmental Protection Agency, Water Quality Specialist
1981-1985

**EDUCATION:**
Bowling Green State University, Environmental Science

Stephen F. Austin State University, MS in Aquatic Biology

Wittenburg University, BS in Biology and Geology

**ORGANIZATIONS:**
Academy of Certified Hazardous Materials Managers

National Association of Environmental Management

**CERTIFICATIONS:**
Certified Hazardous Materials Manager (Masters Level)

Registered Environmental Assessor (REA)

Certified Asbestos Inspector and Management Planner (expired)

Mr. Lee has over 24 years of experience in the investigation, evaluation and remediation of environmental conditions, with a focus on development and redevelopment of impaired properties (Brownfields) and natural resources. He is involved in strategic redevelopment planning and execution, including the procurement and execution of state and Federal grants and loans, site characterization and regulatory closure, investigation and remediation of soil and groundwater contamination. His experience also includes a wide range of land-related environmental services such as due diligence, management and technical responsibility on environmental investigations and remediation, ecological and biological studies, industrial compliance audits, and environmental impact statements. In particular, he is active in assisting communities and developers in processing public and private properties through the Illinois Site Remediation Program and Brownfield Program, utilizing private and public funding.

## PROGRAM EXPERIENCE

**Environmental Site Assessments for Property Transactions** - Involved in ESAs for over a decade and developed and led a global consulting firm's corporate Environmental Assessment Group for 5 years. Has represented both buyers and sellers, and worked with numerous lenders. Involvement has included small to large properties, and simple to complex industrial/manufacturing facilities. He has also been involved in numerous multi-site assessments and corridor studies. Commonly represents larger industrial clients in sale or purchase of multi-facility companies, assisting in price and liability negotiations.

**Brownfield Redevelopment** - Primary role is assisting public and private sector clients in the redevelopment of Brownfield properties. This includes strategy planning, grant processing, and negotiations related to regulatory closure. Has successfully completed U.S. EPA and IEPA Brownfield Grant applications, and has completed numerous projects under state regulatory programs throughout the U.S. In the State of Illinois Mr. Lee has completed projects under the Site Remediation Program, Leaking Underground Storage Tank Program, and Brownfields Program. He maintains close relationships with agency management and staff, and is current on regulatory developments. He has

participated in numerous site closure negotiations with the U.S. Dept. of Justice and the Illinois Office of the Attorney General.

**Environmental Impact Statements and Assessments** - Long history of involvement related to the investigation of soil and groundwater issues. Has participated in EISs and EAs related to the proposed construction of regional wastewater interceptors, airports, and transportation. Has planned and supervised groundwater monitoring programs and developed and implemented soil boring investigations. Has also conducted investigations and assessments related to asbestos, radon, and lead-based paint. Mr. Lee has coordinated environmental assessments and baseline surveys of U.S. Military Installations. He is often called upon to provide expert testimony related to site issues. This activity typically includes a review of site-specific data, and evaluation of contractor performance and site conditions, as related to the applicable regulatory framework. Such reviews are typically performed to evaluate previously implemented remediation technologies, proposed additional investigation or remediation activities, or adequacy of work.

**Site Remediation and Structural Renovations** - Assisted clients in the development of remediation programs to address numerous types of environmental issues, and has managed the execution of remediation projects. He has participated in the investigation and development of remedial alternatives for spill and disposal areas (lagoons, wastewater discharge ponds, etc.). With regard to underground storage tanks, he has supervised removal and abandonment projects ranging from small interior tanks to buried railcars. Mr. Lee has also managed the environmental aspects related to structural upgrading such as bridge and water tower renovation.

**Risk Evaluation/Risk Assessments** - Has been involved in evaluating risks related to environmental impact, and providing associated cost assessments (current and future). Commonly evaluates degree of risk and associated cost magnitudes. Has assisted property owners in successfully addressing environmental concerns prior to marketing of their site. Acts as an agent for owners, meeting with and responding to the interests of prospective lenders regarding properties, which are in various stages of investigation or remediation. Has coordinated performance of full-scale risk assessments to determine the level of risk posed by existing site conditions according to U.S. EPA and state protocols. He is well versed in the completion of Risk Based Corrective Action (RBCA) protocols. He is knowledgeable of the Illinois TACO (Tiered Approach to Cleanup Objectives) protocols, and has obtained state closure approval at numerous sites using this approach.

**Building Investigations** - Mr. Lee has managed projects related to the investigation of buildings and structures. In this regard he has supervised the investigation, inventory, removal and/or repair of asbestos-containing materials, PCBs, lead-based paint, and drums of unknown materials. As a certified asbestos inspector, Mr. Lee has performed asbestos surveys of large industrial complexes.

# R. DAMON LEE, CHMM, REA

*Director of Environmental Redevelopment*

**Ecological/Water Quality Management & Planning -** Assessed environmental impacts of a wide range of engineering and development activities on many different types of water bodies and environments. His activities in water quality management include wetland impact studies, benthic surveys, WWTP effluent impact on receiving streams, septic system influence on lake water quality, and various investigations and assessments concerning developmental impacts on water resources. As a Water Quality Specialist with the Ohio Environmental Protection Agency, Mr. Lee was the Water Quality Plan Coordinator for three of the state's regional planning agencies.

**U.S. EPA Superfund -** Has managed six Superfund sites through the remedial investigation stage and has completed two feasibility studies. His management capacity included negotiation with PRPs and state/federal agencies, and participation in community relations programs.

# R. DAMON LEE, CHMM, REA

*Director of Environmental Redevelopment*

**PROJECT INVOLVEMENT**

**Brownfield Redevelopment**

- Currently providing technical assistance and strategic planning assistance to the Village of Summit, Illinois, relative to local Brownfield sites. V3 prepared and won an IEPA Brownfield Grant for the Village and is preparing to execute project activities that center around redevelopment of a former gasoline service station and grocery store. V3 is also assisting in the Village in redevelopment planning related to a large (30 acre) former scrap yard that is currently under litigation.

- Assisted the Village of Franklin Park in preparing and winning an IEPA Brownfield Grant relative to redevelopment of two blocks of the central downtown district, which includes a former dry cleaning operation. V3 is currently executing Phase I ESA activities in preparation for soil and groundwater investigation of the area.

- Working with Village of Bedford Park in identifying properties relative to IEPA Brownfield Grant and potential interest by local developers.

- Working with the Nicor/ComEd Brownfield Partnership, which is assisting communities identify, characterize and advertise local Brownfield properties. Similarly, he is active with Cook County *Enterpriz*, the redevelopment organization for the county.

- V3 representative in the Brownfield Redevelopment Alliance – which includes V3, Opus Corporation and the Chicagoland Redevelopment Institute. Opus Corporation is one of the largest developers in the US, and is actively seeking Brownfield sites for purchase and redevelopment. The Chicagoland Redevelopment Institute is an affiliate of the Delta Institute, and is a non-profit organization. The Alliance provides value to communities by providing options and wide-ranging expertise in both private and public sector redevelopment.

- Working with the City of Gary, Indiana in the execution of a USEPA Brownfield Grant related to redevelopment of the J-Pit – a former gravel quarry within the city limits. V3 has worked with the Gary Dept. of Environmental Affairs and the Northern Indiana Center for Land Reuse in defining wetland jurisdiction at the site, identifying potential redevelopment options, reviewing site data, and developing a comprehensive investigation scope. V3 anticipates assisting in the quarry reclamation effort through our ecological services group.

- In 2000, V3 prepared a USEPA Brownfield Pilot Site Grant application for the Northwest Municipal Conference. V3 had developed an approach to leverage USEPA funds by obtaining a complementary IEPA Brownfield grant. The grant application was scored high by USEPA, but was not awarded due to demographics of the Conference area.

- Lead V3's partnership with the Chicagoland Redevelopment Institute relative to the purchase, redevelopment and construction of a new building by a large Chicago non-

profit organization.

- Working with the Gary Urban Enterprise Association in the evaluation, planning and design related to redevelopment of 500 acres of mixed wetland and industrial land, to construct a new light industrial park. It is anticipated the park will consist of a number of "pod" developments that integrate the wetland areas, creating an eco-industrial park.

- A global mail provider needed a new central distribution facility in the downtown Chicago area, and wanted to take advantage of the Illinois Brownfield process and assist in redevelopment of an abandoned property located on Goose Island. The property had a long industrial history and significant soil and groundwater contamination. A TIF district was created for the project, and remediation was performed to achieve industrial risk-based closure standards and regulatory closure was completed under the Site Remediation Program.

- One of Chicago's oldest manufacturing companies needed room for expansion, and an adjacent site was identified as suitable. Investigation disclosed significant pockets of contamination. The closure process included coordination with the City of Chicago Department of Environment and their consultants.

**Regulatory Closure**

- When a municipality initiated development activities on a property it had been given a decade ago, soil and groundwater contamination was identified across the site. V3 is working with the three historical site owners (global manufacturing companies) and the municipality, in a partnership, to investigate and remediate the property under the Illinois Site Remediation Program.

- Due diligence disclosed impact under a site building, due to release from battery charging operation. Because the buyer had contracted with contractors, resolution was critical for keeping owner from incurring damages. V3 staff Investigated the area through soil borings, and performed excavation and disposal of impacted soil. A risk assessment was performed to achieve regulatory closure. The project was completed within 6 months.

- Property transaction involving former disposal area lying along back side of site building, where solvents had been dumped for several years. Client (banking institution) has taken title to property as foreclosure, and Phase I Environmental Assessment had not disclosed this problem. Assisted bank in entering Voluntary Program, evaluating nature of impact, and assigning fiscal responsibility. As result, potential buyer accepted title with agreement that closure would be obtained under Voluntary Program. V3 performed remediation and completed risk assessment to

satisfy IEPA that remaining residuals pose no threat.

- During due diligence related to property transaction, soil and groundwater impact was identified under the site building, related to the presence of a former degreaser pit. V3 staff performed a subsurface investigation and delineated the extent and magnitude of impact. Currently working with the seller, pervious tenants, and lenders in developing preferential remedial options. This includes costs, time to remediate, and future liabilities. Additionally, V3 staff has assisted in allocating share of responsibility for the impact, based on tenant occupancy and operations during the history of the site. The site is entered in Illinois Voluntary Program.
- A major Illinois industrial facility has historically operated sludge drying beds as part of its wastewater treatment system. RCRA closure was required for several hazardous waste storage areas at the site, and U.S. EPA has requested that the older lagoons be included in this closure. V3 staff has been assisting the site owner in performing closure under the Illinois Site Remediation Program, thereby avoiding the greater costs and level of effort required under RCRA. This has included negotiating with the IEPA, and keeping current on pending regulations, which could result in the hazardous classification of some sludge being downgraded to special waste.

- Removal of multiple leaking underground storage tanks under state LUST program, and abandonment-in-place of interior tank. Coordinated removal of four 10,000 gallon gasoline and fuel oil USTs, and remediation of impacted soils in tank area. Assisted property owner in obtaining closure of site under state LUST program. Obtained approval for in-place abandonment of interior UST (under plant floor) and performed closure activities.

**Environmental Site Assessments (ESA) for Property Transactions**

- Represented seven-facility, national industrial firm in acquisition by Fortune 100 international corporation. Performed complete due diligence evaluation including compliance, historical issues, and site investigations. Brought all facilities into compliance within four-month period, including all permit applications and operational issues.

- Multi-site environmental assessment related to purchase of 54 communication facilities. Coordinated review of environmental reports pertaining to 54 different operations, to provide potential purchaser with opinion regarding environmental conditions. All properties were placed in three categories: high, moderate, and low risk. Cost estimates for investigating and remediating potential issues were provided for each risk category. Based on this information, the client was able to evaluate the potential risks associated with the proposed purchase, from both an environmental and fiscal standpoint.

- Evaluation of lead contaminated soils at historical gun club. Supervised the field investigation to determine extent and concentrations of lead contaminated soil at property, and assisted client in development of remediation options prior to

marketing of property. Assisted property owner in negotiations with prospective buyers.

- An emerging aerospace industry had identified a competitor as a strategic acquisition regarding growth. One of these firms' facilities was located within a major groundwater contamination plume, and the facility was a potential contributor to the plume. The site was investigated and determined to be a primary source area for the groundwater contamination. Because of the importance of the facility to the client, they remained interested in the acquisition, if the potential financial liabilities could be accurately defined. Costs associated with legal fees, consulting, remediation, and regulatory closure activities were identified within the range of $3.3m to $7.2m, with a most likely scenario of $4.7m. The client negotiated practical terms with the seller and the sale was completed.

- V3 staff's Phase I Assessment indicated the potential presence of a buried discharge lagoon at a Michigan manufacturing facility which was involved in a sale. A soil boring program was performed across the site, using careful field screening techniques to identify and delineate any impact area noted. An approximately 2 ft thick layer of significantly contaminated soil was identified at a depth of 6 ft below ground surface over an approximately 30 ft by 30 ft area. Due to the location of this impact area, this portion of the site was not included in the transfer, and the sale was completed.

**Environmental Impact Statements and Assessments**

- Project Scientist for assessment of potential environmental impacts associated with wide range of development scenarios, including construction and/or modification of roadways, bridges, airports, industrial and commercial buildings, business parks, golf courses, parks, etc. Responsible for project execution according to rigid Federal requirements.

**Site Remediation and Structural Renovations**

- Supervisor/Site Safety Officer for decontamination of New York City Transit Authority electrical station. Building was fully decontaminated for mercury contamination.

- Removal and remediation of multiple hazardous and non-hazardous sludge lagoons. Evaluated regulatory issues and options available for obtaining IEPA closure, and developed and implemented sampling strategy to assess nature of sludge and impact to underlying soil. Performed detailed evaluation of hazardous and non-hazardous disposal facilities within a 200 mile radius. Provided client with detailed cost estimate for removal, transport and disposal of sludge and impacted soil, and options for obtaining closure from IEPA. Prioritized landfills on basis of current and future liability.

- Investigation and evaluation of health risks associated with proposed repainting of municipal water tower located in south central Michigan. Activities included collection of surface paint from tower areas, analysis of material for lead and other metals, and risk assessment of potential public health impacts associated with sandblasting of tower, which was located in a residential area. Evaluation included literature survey, assessment of potential toxicity to identified receptor groups, impacts of sand blasting on paints and resulting distribution factors, and review of studies performed on similar activities and their impacts. Provided expert witness testimony, representing client who had been contracted to do work without being informed of potential toxic characteristics of paint.

**Risk Evaluation / Risk Assessment**

- NASA Lewis Research Center – Cleveland, Ohio and Plum Brook Station. Performed resources base-line evaluation and assessment of environmental impacts of NASA facilities to determine impacts of operations on surrounding habitats.

- State of Wisconsin Hazardous Materials and Waste Survey, to assess State's overall involvement with hazardous materials and waste and identify liabilities. Program included chemical laboratories, transportation operations, fish hatcheries, sign manufacturing shops, hospitals, university buildings, etc. Developed operation-specific questionnaires and created and implemented training program for state personnel. Utilized database for data manipulation. Program enabled state to identify and prioritize environmental issues, and develop cost-effective, systematic approach for addressing multiple facilities.

- Performed assessment of impacts related to proposed renovation of historical bridge in Brownsville, PA. Bridge functioned as main traffic artery to downtown area. Study included evaluation of environmental and socio-economic factors related to renovation. Performed historical research and requested inclusion of bridge on Pennsylvania List of Historical Bridges, which subsequently provided additional funding for upgrade activities.

- Project Manager responsible for drum sampling disposal program associated with active industrial complex in central Ohio. Coordinated staging, field screening, sampling, analysis and disposal of approximately 330 drums containing unknown wastes. Performed liaison with state to avoid action against client.

**Building Investigations**

- Performed sampling of multiple abandoned municipal buildings for lead paint, PCB contamination of wood and concrete floors, metals in dust, and asbestos.

Investigation was performed to determine suitability of buildings for planned renovation activities. Sampling crew of 4 persons over 6 day period.

- Conducted sampling of 40 year old, 400,000 sq. ft. building to determine presence of lead-based paint and asbestos.

- Supervised 2 week sampling and decontamination program related to PCB-contaminated floor areas within historical industrial building.

- The site walkthrough disclosed the potential for PCB contamination at a variety of areas, which was confirmed by later sampling and analysis. The client was provided with cost estimates for additional investigation and remediation of the identified areas, and an inspection plan was prepared according to U.S. EPA guidelines. Based on the probable magnitude of the problem, the client withdrew its letter of intent for purchase of the property.

- Project Manager for asbestos evaluation program at major automotive manufacturer located in Detroit, Michigan. Project was a pilot program which included development of sampling plans and sampling of potential ACM within three buildings totaling 400,000 square feet. Objectives of the study included the development of a cost-effective sampling program to define extent and condition of ACM, assess and prioritize need for abatement actions and provide recommendations and options available, and associated costs. Videotaping was performed to document sampling locations and condition of material. Information collected was entered into a computerized database (dBase III Plus) to allow efficient usage and manipulation, and incorporation into clients computer aided design (CAD) system.
- When Midway Airport closed, a primary airline dissolved its operations, and wastes ranging from solvents, cleaners, paint wastes, lubricants and motor oil were left at the site in containers and drums. V3 staff sampled and characterized these containers, and segregated them by waste stream. Necessary permits were obtained, and materials were appropriately transported and disposed.

- Multi-site project involving 40 warehouse facilities across the United States. Buildings varied from approximately 100 years old to recent, and included extensive variety of warehoused goods, ranging from empty beer cans to hazardous materials. Because of critical time element, client required completion of project within six weeks, and daily progress reports. Project included coordination of seven staff members.

- Drilling Supervisor and Site Safety Officer for field investigation of former MGP site location in downtown Milwaukee, WI. The site was developed as an MGP approximately 150 years ago, and all structures were now located below existing buildings and roads.

**Ecological / Water Quality Management and Planning**

- Field Crew Team Leader for water quality investigation of Lake Murvail and Trinity

River in Texas. Developed sampling program to assess changing ecological conditions in newly created lake. Selected and established physico-chemical and biological sampling stations to perform long-range monitoring of lake conditions. Supervised field crew in performance of water quality sampling along entire length of river. Program included scheduled weekly sampling, and monitoring of low flow and flood conditions.

- South Branch Shiawassee River Remedial Investigation and Feasibility Study, Howell, Michigan. U.S. EPA Superfund. Investigation of 8-mile reach of PCB-contaminated river. Project included evaluation of river dynamics through computer modeling, surface and subsurface soil sampling along river course, collection and analysis of biota (flora and fauna) to perform risk assessment, and development of recommendations for remediation activities.

- Water Quality Assessment related to impacts of construction of proposed large sanitary sewer interceptor on major river in Cleveland, Ohio. Supervised collection of water and biota from 12-mile course of river. Work included detailed analysis of historic and current flow records, and projection of future conditions, and assessment of environmental impacts of existing WWTP and septic system effluent discharge.

- Developed inventory of environmental resources of Bayou La Batre, Alabama, with emphasis on water resources. Worked with regional planning agencies in developing Water Quality Management Plan to provide for protection and enhancement of natural resources.

- As Water Quality Specialist with the Ohio EPA, involved in impact and assessment activities related to on-site wastewater treatment, groundwater protection and water quality management. Functioned as Water Quality Management Plan Coordinator for 3 of State's regional planning agencies. This included monthly meetings with planning agency and city officials, tracking of plan milestones, and interface with OEPA program directors.

**U. S. EPA Superfund**

- Project Manager for Wash King Laundry Remedial Investigation and Feasibility Study, Baldwin, Michigan. U.S.-EPA Hazardous Waste Site. Investigation of groundwater contamination associated with PCE release from dry cleaning operation. Project included definition of groundwater contaminant plume, assessment of impact to receiving stream, and characterization of source areas.

- Project Manager for West Michigan Avenue Remedial Investigation and Feasibility Study, Oshtemo, Michigan. Michigan 307 Hazardous Waste Site. Investigation of groundwater contamination associated with improper waste disposal by multiple industries. Program included sampling and analysis of soils and groundwater throughout industrial area to identify and characterize individual contributors.

- North 34th Street Remedial Investigation and Feasibility Study. U.S.-EPA Hazardous Waste Site. Investigation of groundwater contamination associated with historical industrial operations. Program included sampling and analysis of multiple aquifers.

- Electro-Voice Remedial Investigation and Feasibility Study. U.S.-EPA Hazardous Waste Site. Soil and groundwater investigations related to historic wastewater discharge to on-site lagoons and on-site underground storage tanks.

- Conducted monitoring of sanitary wastewater sludge for use in land application (non-crop) strategy related to use in sod farms. Supervised sampling and analysis, and completed coordination with USEPA representatives.

# THOMAS E. SLOWINSKI

*Principal/Director of Wetland/Ecology Group*

**PROFESSIONAL EXPERIENCE:**
V3 Consultants,
Principal/Director of
Wetland/Ecology Group
1997-Present

The S/E Group, Vice President
1989-1997

U.S. Army Corps of Engineers,
Chicago District, Chief,
Regulatory Functions Branch
1984-1989

U.S. Army Corps of Engineers
Biologist, Regulatory Functions
Branch
1983-1984

U.S. Army Corps of Engineers
Biologist, Environmental and
Social Analysis Branch
1976-1983

**EDUCATION:**
Governors State University,
University Park, Illinois, Master
of Arts, Environmental
Management

Marquette University, Milwaukee,
WI, Bachelor of Science, Biology

**PROFESSIONAL CERTIFICATIONS:**
Certified Wetland Specialist, Lake
County Stormwater Management
Commission (2002).

Qualified Wetland Review
Specialist, Kane County
Department of Environmental
Management (2002).

**PROFESSIONAL ASSOCIATIONS:**
Society of Wetland Scientists
Home Builders Association of
Greater Chicago

Mr. Slowinski has 25 years of wetland and environmental impact assessment experience. He directs the activities of an interdisciplinary staff of botanists, soil scientists, biologists, and ecologists in performing wetland, ecological restoration and environmental impact assessment services. These services include wetland delineation and assessment, wetland mitigation planning and design, U.S. Army Corps of Engineers Section 404 and local permitting, wetland management and monitoring, ecological restoration projects and the preparation of environmental impact assessments. Mr. Slowinski provides consultation on all environmental and regulatory issues and provides expert testimony regarding wetland and other environmental issues.

## PROJECT INVOLVEMENT

### V3 Consultants

**Wetland Delineations.** Supervised or participated in more than 1,000 wetland delineations in accordance with the appropriate Federal wetland delineation manual in the Chicago metropolitan area, St. Louis area, Indiana and Wisconsin. This included the delineation of over 100 wetland areas within the 4,000-acre study area of the Lake County, Illinois extension of Route 53 (FAP 342).

**Corps of Engineers and DuPage County Permit Applications.** Responsible for preparation, submittal and coordination of approximately 400 permit applications, including development of strategies to obtain permits in a timely manner.

**Wetland Mitigation Design.** Supervised mitigation planning and design for more than 300 projects requiring Section 404 permits.

**Wetland Management and Monitoring.** Supervised preparation of and implementation of wetland management and monitoring plans as required by Corps of Engineers Section 404 permits, including prescribed burn management of wetlands.

**Ecological Restoration.** Project Manager and primary contact for V3's Professional Services Agreement with City of Chicago, Department of Environment for Professional Ecological Analysis, Restoration and Interpretation.

- Nature Center/Nature Preserve Feasibility Study, May 1997. Study won the 1998 American Society of Landscape Architecture,

Illinois Chapter President's Award for Landscape Planning and Analysis.

- Indian Ridge Marsh Environmental Center Feasibility Study, July 1999.
- Calumet Area Ecological Management Strategy, July 2001

**Environmental Planning.** Nahant Marsh Master Plan, Davenport, Iowa for River Action, Inc. and Nahant Marsh Steering Committee, October 1998. Project Manager for development of master plan for 500-acre Nahant Marsh, an important urban wetland. The master plan evaluated the preservation, protection and enhancement opportunities for the privately owned marsh and surrounding area.

**FAP 342, Lake County Extension of Route 53/I-355, Chairperson, Illinois Department of Transportation Wetland and Water Quality Technical Committee, 1996.** This committee consisted of technical experts on wetland, surface water quality and groundwater quality, and was formed to provide input on the scope of required technical studies, preparation of technical reports, impact assessment, wetland mitigation requirements and site selection, and to assist with agency and municipal coordination on all wetland and water quality issues.

**U.S. Army Corps of Engineers, Chicago District**

Chief, Regulatory Functions Branch. Responsible for planning, supervising, directing and coordinating all technical and administrative functions of the Section 404 Regulatory Program at the Chicago District, U.S. Army Corps of Engineers. Responsible for final recommendations to District Engineer on all policy and permit decisions. Involved in Section 404 permit requirements including interagency and public coordination, and preparation of Environmental Impact Statements for the following controversial permits: Waste Management, Inc. landfill, Lake Calumet, Chicago; Illinois Department of Transportation Hickory/Spring Creeks flood control project, Joliet; proposed 1992 Chicago World's Fair, Chicago; and, U.S. Environmental Protection Agency Superfund Project, Waukegan Harbor.

**North-South Tollway, DuPage County, Illinois Section 404 Permit.** Project manager with final responsibility for all interagency and public coordination, scope and preparation of Draft and Final Environmental Impact Statements, and development and construction monitoring of wetland mitigation plan.

**Environmental Studies and Impact Assessment.** As a senior staff member of the Environmental and Social Analysis Branch, responsible for the organization and direction of an interdisciplinary environmental staff in all aspects of federal water resources projects including collection of environmental data, formulation of alternatives, environmental impact assessment, and the identification and resolution of environmental issues. Projects included Little Calumet River, Indiana Flood Control Project, and various Confined Disposal Facilities and Small Boat Marinas.

**Habitat Evaluation Procedures (HEP)**

Project manager for U.S. Army Corps of Engineers. U.S. Fish and Wildlife Service 1980 Habitat Evaluation Procedures National Demonstration Project, Little Calumet River, Indiana Flood Control

ECT.CON INC.

# Terrie M. Baranek

**Years of Experience:** 18

**Education:**
The American University
M.S. Physical Chemistry
1987

University of Maryland
Institute of Physical
Science and Technology
Chemical Physics
1983-1984

Washington & Jefferson
College
B.A., ACS, Chemistry
1983

CCAC
A.A. Computer Science &
Information Technology
1998

**Contact Information:**
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**General Qualifications**

Ms. Baranek is the President of ECT.CON INC. with experience in the performance of a variety of environmental assessments. Her responsibilities include data validation, data evaluation and management, environmental chemistry. Ms. Baranek has extensive experience in all facets of these technical areas. Examples of her experience in these areas are presented below.

**Experience**

**Data Validation/Chemistry**

- Performed data usability and validation for a variety of projects. Data validation was conducted in accordance with the project-specific QAPP, National Functional Guidelines, State and USEPA Regional requirements including Regions I, II, III, IV, and V, and the analytical method. The following analytical fractions in aqueous, air and solid media were validated: VOCs, SVOCs, Pesticides, PCBs, PAHs, TPH (GRO/DRO), general chemistry parameters (e.g., TOC, TOX, chlorides, nitrates/nitrites, sulfate, sulfides, alkalinity, acidity, TDS, TSS, etc.), metals (including mercury and cyanide), white phosphorous, chemical warfare agents and explosives.

- For a Navy Installation in Indiana, a field investigation for a Dye Burial Ground was to be conducted. In the planning stages, analytical method development for dyes and smoke-related dyes needed to be conducted. Responsible for providing management oversight on the method development to ensure that it meet the method development requirements of the RCRA Program as stipulated in SW-846 Method Development and Performance guidance. Performed data validation of the dye samples.

- Conduct technical audits of analytical environmental laboratories. Review processes to ensure that the laboratory uses Good Laboratory Practices (GLP) and performs activities in accordance with industry standards.

- Prepared a QAPP for an IDEM state-lead Superfund site. The QAPP was developed in accordance with USEPA Region V guidance. The document addressed both on-site (mobile lab) and off-site analytical procedures. Coordinate with laboratories to incorporate their QA/QC information and SOPs.

Project. Involved habitat evaluation of 2400 acres, including 1300 acres of wetlands, and preparation of report.

**Presentations/Copyright**

Baranek, T.M. "Data Validation" Presented at the Water and Environment Federation International Technical Conference, Atlanta, Georgia, October, 2001.

Baranek, T.M. Chairperson for the Environmental Software Showcase Environmental Resources in the Information Age Technical Sessions at the Air and Waste Management Association/Duquense University Co-Sponsored Conference, May 1999.

Baranek, T.M. "Understanding and Using the Data Life Cycle." Presented at the Air and Waste Management Association Western Pennsylvania Meeting, March 1998.

Baranek, T.M. "WILLIE - Welcome to the World of Data Management." Registration No. TX3693681, Dec. 1993.

Baranek, T.M. "Technical and Regulatory Considerations for Environmental Sampling and Analysis Plans." Presented at the American Chemical Society 25th Central Regional Meeting, October 1993.

Baranek, T.M. Chairperson for the Environmental Chemistry and Industrial Chemistry Sessions at the American Chemical Society 25th Central Regional Meeting, October 1993.

Baranek, T.M. and Carfagna, P.F. "Data Management and Risk Assessment." Presented to the Pennsylvania Department of Environmental Resources as a Technology Transfer Seminar, August 1993.

Baranek, T.M. "Sampling Strategies for CERCLA RI/FS Programs." Presented at the PPG Industries RI/FS Technology Transfer Seminar, March 1993.

Baranek, T.M. "The Emergency Planning and Community Right-to-Know Act of 1986." Presented at the 12th Annual Pacific Southwest Health & Safety Seminar, April 1989.

Baranek, T.M. "SARA Title III and Small Companies." Presented at the Carnation Company National Conference, October 1988.

Baranek, T.M. and Waters, P.F. "Slag Encapsulation of Toxic Metal Salts." Presented at ACS Washington, DC Chapter. 1987.

- Project Scientist for the development of a Region V QAPP for an Ohio Superfund site. Attended the Pre-QAPP meeting and prepared the QAPP in accordance with the USEPA Region V CERCLA QAPP guidance. Coordinate with laboratories to incorporate their QA/QC information and SOPs.

- Prepared the Quality Assurance Project Plan (QAPP) for the the correct analytical protocols would be employed to obtain quantitatively usable data. Tracked samples from the field chain-of-custody to receipt from the data validators. Reviewed data and reduced the data set according to EPA guidance. Generated analytical and statistical tables.

Conduct data quality objective assessment to develop cost-effective sampling and analysis plans

- Based on the data quality objectives (DQOs) for the project, prepared numerous field sampling plans to gather information to conduct risk assessment. The plans included the number, type and location of samples. Statistical sampling approaches were used, when appropriate. Identified the analytical parameters to be include in the sampling program. Cost-effective analytical methods (e.g., field testing, mobile lab, CLP, etc.) to meet the DQOs were identified.

Reduce analytical cost by implementing field analytical programs

- Developed a cost-effective analytical strategy for the RFI Phase II investigation at a large aerospace manufacturing facility. Reduced analytical costs while still obtaining quantitative data to delineate horizontal extent of contamination at a SWMU. Used a field UV spec (metals) and field GC (VOCs). Incorporated these data into a GIS system and prepare 3D visual illustrations to assist in determining volume of soil for in-situ remediation.

- Analyzed EPA's regional laboratory's capabilities and role in support of selected RCRA, TSCA, CERCLA, CWA, and CAA programs

- Developed the implementation strategy for the Toxicity Characteristic Leaching Procedure (TCLP). Interviewed affected parties to identify major issues which include notification, education, compliance and monitoring of the regulated community; and preventing laboratory capacity shortfalls arising from the rule. Developed options paper for these major issues.

- Participated in EPA's Region II Environmental Services Division (ESD) Program Review. Developed questionnaires regarding analytical instrumentation; interviewed Program Directors on their section's capabilities; statistically analyzed information; compiled the data; and prepared the review

document. The document highlighted current ESD problems, initiatives for addressing the problems, and opportunities for improving the quality of ESD services.

**RCRA Corrective Action**

Negotiate reduction in field QA/QC samples to reduce program costs

- Senior QA/QC Advisor for the Bethlehem Steel Corporation (BSC) Burns Harbor, Indiana RCRA RFI. Responded to USEPA Region V comments on the workplan/QAPP and further developed the existing workplan/QAPP to enhance the efficiency of the field program. Additionally, conduct field and laboratory audits to ensure adherence of the program to the approved workplan/QAPP. Respond to questions from the staff on clarification of workplan/QAPP intent. Review field and analytical data for accuracy and completeness. Evaluate conclusions drawn from the data for validity.

Participate in USEPA Region 5 Pre-QAPP Meeting and Negotiate Screening Criteria and Analytical Methods

- For a Navy Installation located in Indiana, prepared the Quality Assurance Project Plan (QAPP) for a RCRA field investigation. This QAPP was developed in accordance with USEPA Region 5's 1998 QAPP Guidance and Policy Manual. Development of the QAPP including the analysis and incorporation of historical data. This information was used in the DQO process to develop the sampling plan for the project.

**Regulatory Development and Analysis**

- Developed options for the testing\certification requirements for the reproposed rule making for RCRA's Organic Toxicity Characteristic (OTC).

- Responded to public comments received on the first proposed RCRA OTC regulations.

**Collegiate Instructor** (1998 to 2001)

Instructor in the Environmental Science and Chemistry Departments at the Community College of Allegheny County.

**Continuing Education**

Practical Environmental Sampling and Analysis - ACS
Environmental Data Validation - ACS

SIMALABS
INTERNATIONAL

# Jeffrey M. Loewe
## QA/QC Director

EDUCATION:

B.S., Chemistry, University of Dubuque, Dubuque, Iowa 1987

PROFESSIONAL EXPERIENCE:

Mr. Loewe is responsible for the overall coordination of the Quality Assurance program at the Merrillville Laboratory. He is responsible for the laboratory's accreditations, and ensuring adherence to all aspects of the Quality Assurance Plan. These aspects include documentation systems, analytical compliance, performance samples, training records, and internal audits.

Mr. Loewe has accumulated more than fifteen years of laboratory experience, thirteen of which were in the environmental industry. He has experience as an analyst, in procedural training, laboratory set up, project management, and in various laboratory certification standards.

1999-2001 QA/QC Director-PDC Laboratories, Peoria, Illinois

Responsible for the overall Quality system of the laboratory. Implemented a NELAC compliant QA program, as well as approval by the US Army Corps of Engineers. Responsible for staff supervision and the implementation of a new Laboratory Information Management System (LIMS).

1990-1999 QA/QC Coordinator-Daily Analytical Laboratories, Peoria, Illinois

Responsible for maintaining State certification and the implementation of the Quality Assurance Plan. Served as a project manager and coordinator of a monthly wastewater quality control program. Also responsible for training records and Standard Operating Procedure generation.

1988-1990 Chemist, Daily Analytical Laboratories, Peoria, Illinois

Responsible for various analyses of environmental samples.

1985-1988 Chemist, Midwest Grain Products, Dubuque, Iowa

Responsible for analyses on raw materials and finished product.

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SIMALABS
INTERNATIONAL

# Michelle A. Dilley
## Project Manager

**Education:**
B.S. Biology & Environmental Science, Lincoln Memorial University, Harrogate, Tennessee 1999

**Professional Experience:**
Ms. Dilley, as Project Manager for SIMALABS International, is responsible for the oversight of client projects. Ms. Dilley's responsibility is tracking samples from the time of receipt to the time of reporting the data. She works together with the Laboratory Managers, Sample Custodian, QA/QC officer and reporting staff. She verifies all information for the projects and transfers the information to the appropriate parties which includes but is not limited to the parameters requested, reporting limits, data quality levels, holding times and due dates. She also corresponds with the clients verifying request concerning their samples and tracking progress when requested.

Prior to joining SIMALABS, Ms. Dilley was a Formulation/Synthesis Technologist for AOC in Collierville, Tennessee. Her responsibilities included conducting complex experimental procedures for new product development at a leading polyester resin company's Research and Development center utilizing an array of specialty chemicals. Made several technical service customer calls for assigned accounts. Aided in the production scale-up of many new products, requiring copious amounts of hands on experience in a polyester chemical plant environment.

Ms. Dilley has been working in the environmental industry since 2000.

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**Baker Environmental, Inc.**
*A Unit of Michael Baker Corporation*

# Margaret C. James

Environmental Scientist

**Years with Baker:** 4.5
**Years with Other Firms:** 0

**Education**
Saint Joseph's College
B.S., Environmental Science,
1996

## General Qualifications

Ms. Margaret (Peggy) James is an assistant environmental scientist with experience in ASTM Standard Phase I and II Environmental Site Assessments, ecological surveys, and environmental database management, database design, and SARA Title III Compliance Work.

## Selected Experience

**Phase I/II Environmental Site Assessments**

- Performed a Phase I Site Assessment for Lake Erie Land Company, Chesterton, Indiana, prior to property transfer.
- Performed a Phase I Site Assessment for an Industrial area owned by Northern Indiana Public Service Company (NIPSCO), Hammond, Indiana.
- Performed Phase I/II Site Assessments at four locations for The Holladay Group, South Bend, Indiana. Limited soil sampling was performed as a result of the Phase I ESA. Adjacent properties were industrial, commercial, and residential.

Work on ESAs involved: conducting site visits, recognizing environmental concerns (past and present), reviewing records and previous reports, researching property title transfers and historical usages, conducting interviews, making recommendations and writing reports.

**Environmental Sampling Experience**

- Provided RCRA RFI services to a major steel manufacturer in the Midwest. Assisted in the groundwater-sampling program including well development, purging, and sampling as well as hydropunch groundwater sampling. Additional services included environmental database management, analytical data management, ecological field survey, and soil and waste stream sampling. Assisted with laboratory data validation management and QA/QC activities.
- Provided RCRA RFI services to a major steel manufacturer in the Northwest, Indiana. Performed four quarterly-rounds of groundwater sampling at 55 monitoring well locations. Groundwater sampling program included well development, low flow purge, collection of field parameter including, pH, specific conductivity, dissolved oxygen, oxidation-reduction potential, turbidity, and temperature.
- Performed hazardous waste characterization sampling to a major steel manufacturer in Northwest, Indiana. Collected several different waste stream samples for various analyses. Samples were then labeled and documented for Chain of Custody's and shipped offsite to an

independent laboratory.

- Performed surface water and sediment sampling for a major steel manufacturer in Northwest, Indiana. Sampling included collection of surface water and sediment samples for several depths. Samples were then labeled and documented for Chain of Custody's and shipped offsite to an independent laboratory.

- Performed surface soil sampling for a manufacturing facility in Central Indiana. Sampling consisted of eight riverbank locations using hand augers and stainless steel spoons. Samples were then labeled and documented for Chain of Custody's and shipped offsite to an independent laboratory.

- Performed groundwater sampling at a chemical manufacturing facility in Charleston, West Virginia. Sampling consisted of twenty-four flush mount wells using tephlon bailers. Samples were then labeled and documented for Chain of Custody's and shipped offsite to an independent laboratory.

- Performed groundwater sampling for a manufacturing facility in Central Indiana. Sampling consisted of thirteen flush mount monitoring wells at a low flow purge. Field parameters were collected on sight using Horiba U-22 model and HACH total iron field kit. Samples were then labeled and documented for Chain of Custody's and shipped offsite to an independent analytical laboratory.

- Performed groundwater sampling for a major steel manufacturer in Northwest, Indiana. Sampling consisted of sixteen flush mount and standpipe monitoring wells by either low flow purge or tephlon bailers dependent upon site conditions. Field parameters were collected on sight using Horiba U-22 model, YSI meters, and HACH total iron field kit. Samples were then labeled and documented for Chain of Custody's and shipped offsite to an independent analytical laboratory.

**Database Management**

- Provided RCRA RFI services to a major steel manufacturer in the Midwest. Maintained a GIS database containing well construction data, geologic information, groundwater elevation measurements, and survey data. Developed several Microsoft Excel spreadsheets encompassing topics of sample tracking, invoice tracking, laboratory analytical tracking, groundwater elevations, and analytical summary tables. Additional services included environmental database management, analytical data management, ecological field survey, and soil and waste stream sampling. Assisted with laboratory data validation management and QA/QC activities.

- Analytical Data manager to a major steel manufacturer during the coarse of a RCRA corrective action being performed in Northwest, Indiana. Responsibilities include data tracking of many different sampling activities occurring at various locations at the facility. Create and maintain a sample-tracking database that encompassed sample collection information, relevant field parameters, information and chain of custody information. Create and maintain a unique identifier system to correspond with each sampling location and depth for various sample media including surface soil, subsurface soil, surface water, waste, groundwater, and sediment. Create and maintain uniform field parameter reporting spreadsheets. Facilitate smooth accurate transfer of sample from field locations to laboratory under proper chain of custody and assurance that all necessary analysis is performed for any given sample. Transfer electronic laboratory data and validation of that data from text format for final input into Terra Base.

- Provided data management service for Bayer Corporation, Elkhart; Indiana. Assisted in developing several Microsoft Excel spreadsheets and Microsoft Access Databases encompassing topics of equipment tracking, waste disposal activities, hazardous waste manifests, and tracking of asbestos removal activities.

- Provided data management service and research knowledge to a major steel manufacturer in Northwest, Indiana. Created and maintained Microsoft Access and Excel spreadsheets to assist in the development of SARA 312/313 reporting documents for the facility. Performed field inventory to collect necessary information to populate the database.

- Provided data management service and research knowledge to a major steel manufacturer in Northwest, Indiana. Created and maintained Microsoft Excel spreadsheets to assist in the development of a conceptual site model detailing spills and releases throughout the facilities operation. Assisted in the development of a comprehensive listing of areas of environmental interest contained within the facility.

- Provided data management service for the Indiana Department of Transportation (INDOT). Developed databases to assist in the collection and coordination of efforts during a relocation study being performed in the Lafayette, Indiana area. Databases include an administrative filing tracking system for project related documents and a Public Comments database.

- Provided extensive research of State of Indiana, Federal and local sources for a confidential client in Indiana. The research was conducted on an accelerated schedule for ongoing negotiations and later mediation/litigation support. Research included the review of Resource and Recovery Act (RCRA), Office of Solid and Hazardous Waste (OSHW), Department of Natural Resources (DNR) Water and Fish and

(OSHW), Department of Natural Resources (DNR) Water and Fish and Wildlife, Army Corps of Engineers, USEPA, IDEM, VRP, Spill, UST, LUST and Community Right To Know data bases, Newspaper, NPDES, Stormwater, Office of Water, Formerly Used Defense Sites (FUDS) and local library files for more than 10 large manufacturing facilities and an industrial corridor. Reviewed the files for specific key data to support ongoing negotiations. Data was sorted, indexed and assembled in a cross-referenced database as an aid for potential litigation.

- Assisted in preparation of a Spill Prevention Control and Countermeasure Plan for a petroleum retail facility in Lake Station Indiana. Performed record searches to determine whether or not the facility was in compliance with the guidance set forth by the United States Environmental Protection Agency (USEPA) Region V.

**Database Design**

- Designed and implemented a Microsoft Access database to assist in tracking and responding to public comments from agency, local government, and public sources for Indiana Department of Transportation Route 231 Relocation project.

- Designed and implemented a Microsoft Access database to assist in the tracking of project related documents for the Indiana Department of Transportation Route 231 Relocation project.

- Designed and implemented a Microsoft Access database to assist in tracking Fugitive Dust sources at a major steel manufacturing facility in Northwest Indiana. Database was used in compiling fugitive dust sources, information regarding vehicular traffic, and road conditions which contribute to fugitive dust releases.

**SARA Title III Compliance Work**

- Performed and prepared SARA 312 compliance services for a large steel client's facility in Northwest Indiana. These services involved compilation of data, conducting chemical inventories, determining toxic release inventories. These services were performed to ensure SARA Title III, Emergency Planning Community Right-to-Know compliance.

**Related Courses**

- *Amphibian Research Project I & II*: Studied the amphibian populations in Jasper County, Indiana in order to detect whether these populations are declining. *Amphibian Research Team Leader, 1996*

- *Ichtheology & Herpetology:* Learned identification and field techniques involving reptiles and amphibian species.

- *Ornithology & Mammology:* Learned identification and field techniques involving bird and mammal species.

**Computer Skills**

- Microsoft Access
- Microsoft Excel
- Microsoft Word
- Microsoft FrontPage 2000
- Microsoft Office '97 '00
- Microsoft Project
- Microsoft PowerPoint
- WordPerfect
- GIS Scout
- Windows 3.1/Windows '95, '98
- Terra base
- Visio

**Certifications**

- 40 hour 29 CFR 1910.120 Haz/Waste Emergency Response Program, 1997.
- 8 hour 29 CFR 1910.120 OSHA Refresher Training, 2000.
- 38 hour U.S. Army Corps of Engineers Wetland Delineation Certification Training Program, 1998.
- 24 hours Initial Asbestos Building Inspector, 2001.

**Continuing Education/Seminars**

- Introduction to Microsoft Access 97 for Windows, Purdue University Calumet, September, 1999.
- Access 97 for Windows: Intermediate Level, Purdue University Calumet, November, 1999.
- Elementary Methods for Statistics, Purdue University Calumet, May, 2000.
- What You Need to Know About the New Wetland Regulations, J.F. New & Associates & Plews, Shadley, Racher, & Braun, May 2000.